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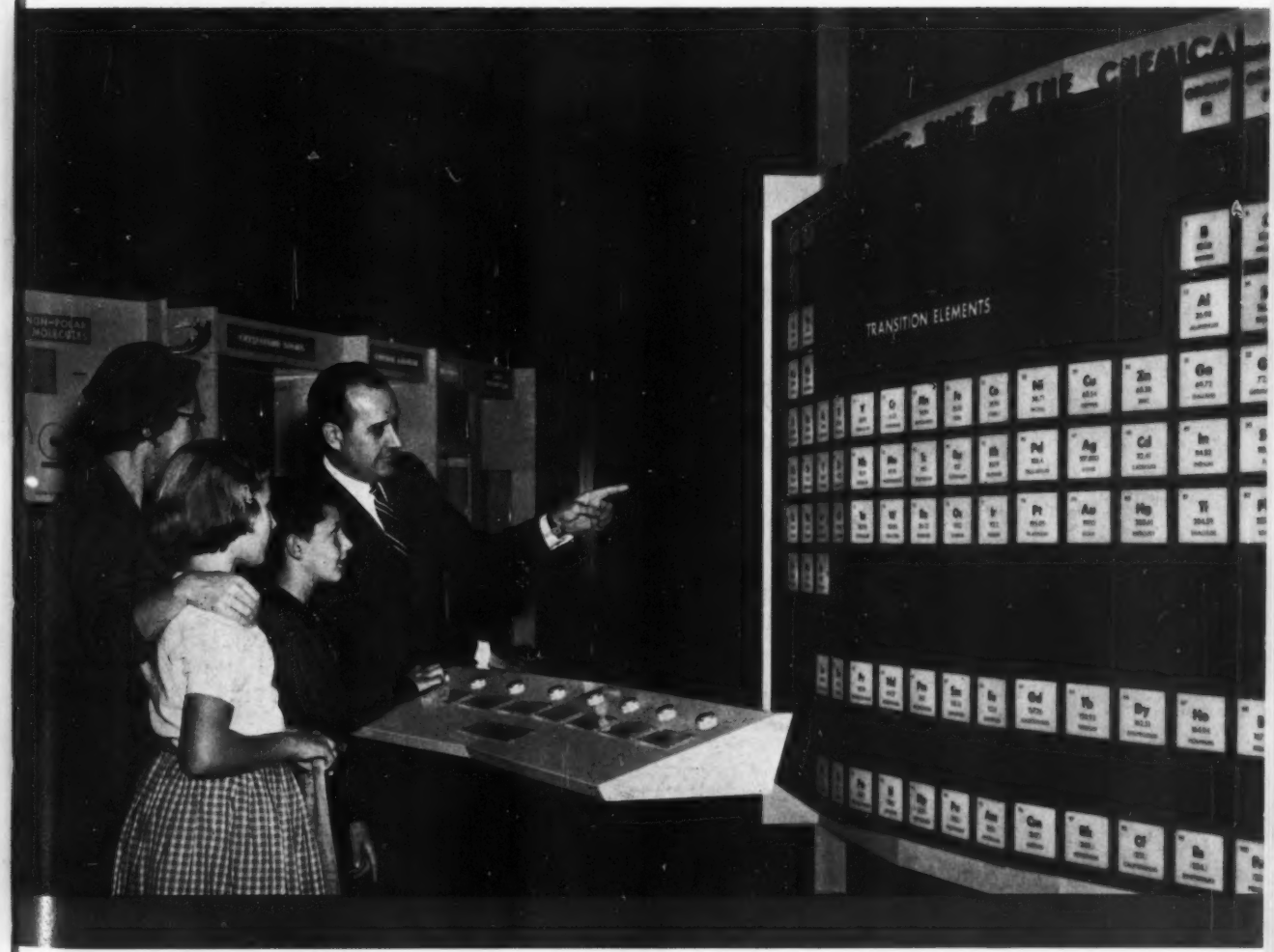

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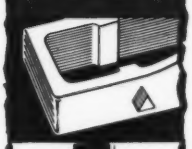

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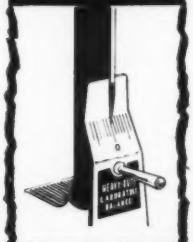

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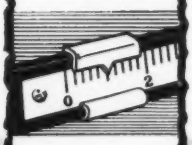

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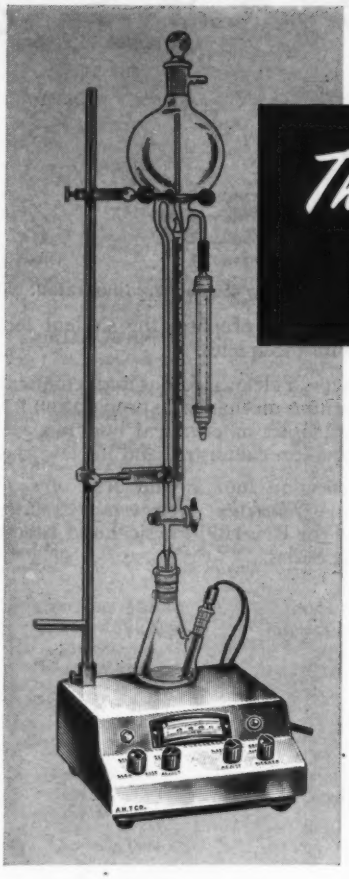

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Vol. 38 / No. 12

590 The Nature of Metal-in-Amine Solutions, *E. Charles Evers*

597 The Franklin Institute Chemistry Room: A Design for Learning, *Irwin Siegelman* and *Joseph S. Schmuckler*

601 A Model for Optical Rotation, *Leon L. Jones* and *Henry Eyring*

606 Models Illustrating Types of Orbitals and Bonding, *Wilbur L. Baker*

607 A Qualitative Elemental Analysis System Correlated with the Periodic Table, *Ernest H. Swift* and *William P. Schaefer*

611 Determination of Saponification Equivalents of Phenolic Esters, *S. W. Tobey, S. D. McGregor,* and *Sharon L. Cottrill*

614 The Liebig-Pasteur Controversy: Vitality without Vitalism, *George E. Hein*

619 Adapting Hospital "Castoffs" for Use in a Chemistry Laboratory, *Sister Mary Martinette, B. V. M.,* and *Sister Agnita Crowell, D. C.*

620 The Discovery of Germanin by Oskar Dressel and Richard Kothe, *Jutta Dressel,* translated by *Ralph E. Oesper*

622 Aleksei Evgen'evich Chichibabin, *E. Cerkovnikov*

625 International Atomic Weights—1961

626 Device to Convert a Hypodermic Syringe into a Multi-Purpose Apparatus, *Samuel Shapiro*

627 Textbook Errors, 35: Miscellanea No. 4, *Karol J. Mysels*

628 Chemical Information Centers of Latin America, *J. Vikin*

630 Institutional Influences in the Graduate Training of Eminent Chemists, *B. R. Siebring*

632 A Biochemistry Laboratory Experiment, *Irwin Scher, Edward Walsh,* and *Barney Kadis*

633 A Titration Controller for Demonstrating the Behavior of Acid-Base Indicators, *John T. Stock*

634 A Convenient Safety Shield for Small-Scale Apparatus, *Stanley H. Langer* and *Peter Pantages*

634 Melting Point Apparatus, *Carlton S. Graves*

635 Division of Chemical Education of the ACS: Report from the Treasurer, *W. G. Kessel*

636 Report of the New England Association of Chemistry Teachers: Official Business, *Robert D. Eddy*

Facing 641 Notice to Authors

641 Annual Index

Features

589 Editorially Speaking

A905 Out of the Editor's Basket

637 Letters

639 Book Reviews

A933 Advertisers' Index

A895 Chem. Ed. Tested Demonstrations, *Frederic B. Dutton*

A869 Chemical Instrumentation, 15. Gas Chromatographs, *S. Z. Lewin*

A891 Chemical Projects, *Jay A. Young* and *John K. Taylor*

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Chemical Instrumentation

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This series of articles presents a survey of the basic principles, characteristics, and limitations of those instruments which find important applications in chemical work. The emphasis is on commercially available equipment, and approximate prices are quoted to show the order of magnitude of cost of the various types of design and construction.

15. Gas Chromatographs

The field of gas chromatographic instrumentation is unique in the history of science, for never before has a new instrumental technique been so quickly appreciated, exploited, and caused to proliferate. Although gas adsorption chromatography has been known, and sparingly used, since about 1940, it was not until 1952 that the first experimental demonstration of gas-liquid partition chromatography (GLPC) was published by A. J. P. Martin and A. T. James. The first commercial units for GLPC were brought out almost simultaneously by four manufacturers in 1955. Only 6 years later, by mid-1961, the manufacture and sale of laboratory gas chromatographs by more than 20 different manufacturers had become a $4,000,000-per-year industry in the U. S., with no indication that the market for these instruments had reached a plateau. It has been estimated that there were over 10,000 laboratory gas chromatographs in use in mid-1961; this figure may be compared with the corresponding estimate of 5000 infrared spectrometers and 500 mass spectrographs (fields of instrumentation which have been commercially exploited since about 1946). These estimates do not include process control instruments.

The term *gas chromatograph* will be used here to designate any instrument in which the moving phase is a gas, and in which a distribution in space or time of the components of a gaseous sample is achieved through any of the chromatographic mechanisms; viz., selective adsorption, partition, or vaporization. If the principal mechanism producing the distribution is adsorption chromatography on a solid substrate, the instrument is commonly called a *gas adsorption chromatograph*. The term gas-solid chromatograph is also used for this type of device. If the principal mechanism involves partition between the gas phase and a liquid film supported on an inert solid, the instrument is often called a *gas-liquid partition chromatograph*. Other terms applied to this type of device are: gas-liquid chromatograph, vapor phase chromatograph, vapor fractometer, vapor partitioner (the last two designations are also used as the trade names of the commercial units made by Perkin-Elmer Corp. and Fisher Scientific Co., respectively).

The gas chromatograph is a remarkable tool. It has been used to separate and identify dozens of constituents in less than a microgram of sample. It has also been scaled up to the preparative level, and instruments have been designed for the purification (to a degree hitherto unattainable) of kilogram amounts of material. It is inherently simple to use, rapid in response, and gives a quantitative, easily interpretable read-out. It is applicable to practically all classes of substances, from the fixed gases, employing columns cooled to low temperatures, to high molecular weight, non-volatile substances, utilizing columns heated to temperatures in excess of 500°C.

GAC and GLPC instruments are basically the same in design; they differ principally with respect to the nature of the column material through which the sample is passed. In general, a gas chromatograph consists of the following components, as illustrated in Figure 1. *A.* Carrier gas source; *B.* Gas flow control and meter; *C.* Sample injection and vaporization chamber; *D.* Chromatographic column; *E.* Temperature program and/or control; *F.* Gas composition detector; *G.* Read-out device.

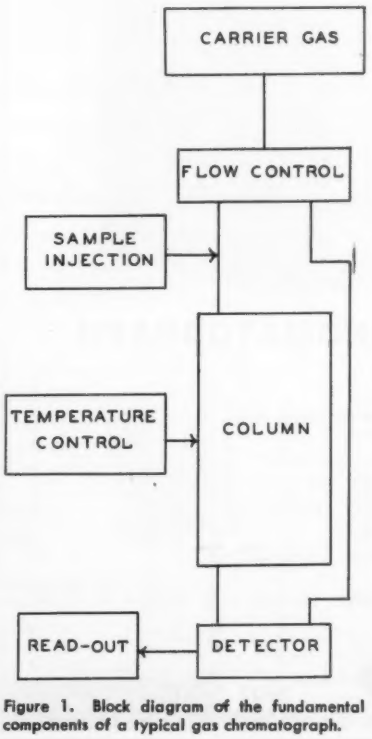

Figure 1. Block diagram of the fundamental components of a typical gas chromatograph.

Although the chromatographic column is the heart of the instrument, and its characteristics set the ultimate limits of performance, the practical results achieved in the use of a given instrument are strongly dependent upon the design of the other components. Hence, it is essential that the properties and limitations of these components, as well as the manner in which their influence is felt on the overall performance, be clearly understood.

Carrier Gas

The carrier gas is the motive agency that sweeps the sample through the column. It must be stable, and available in quantity in high purity; it must interact only weakly with the chromatographic substrate in order not to interfere with the distribution mechanism; and it must not markedly affect the detector in order to permit the latter to sense the sample components as they emerge from the column. Hydrogen, helium, argon, nitrogen, oxygen, and carbon dioxide are the most commonly used carrier gases. Hydrogen and helium are particularly suitable for instruments which employ thermal conductivity detectors, since their thermal conductivities are much greater than those of all other gaseous substances, and hence they permit the best sensitivity in the detection of sample molecules in the column effluent. Argon is uniquely useful in connection with the beta-ray ionization detector (see section on detectors); carbon dioxide is employed in conjunction with the nitrometer detector, in which a sodium hydroxide solution is utilized to absorb the effluent carrier gas, leaving a residue of sample components, the volumes of which can be measured directly. Thus, the chemical nature of the carrier gas has an important effect on the *sensitivity* of a gas chromatograph.

The molecular weight, diffusivity, and viscosity of the carrier gas also have important effects on the *resolution* of the gas chromatograph. At low flow rates of the carrier gas through a packed column, the heavier carrier gases give the best performance, since the disturbing effect of random diffusion in the gas phase is minimized. However, at high flow rates in capillary (unpacked) columns, the light gases are superior, for under these conditions the limiting factor becomes the rate of equilibration between gas and liquid phases, and the light gases diffuse into and out of the liquid substrate faster than the heavier carrier gases.

(*Continued on page A870*)

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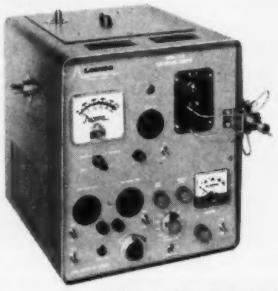

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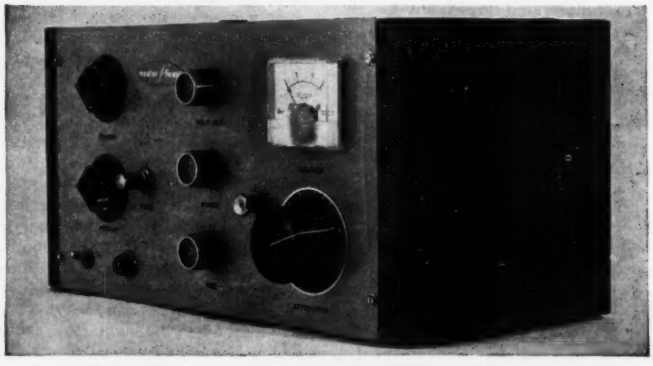

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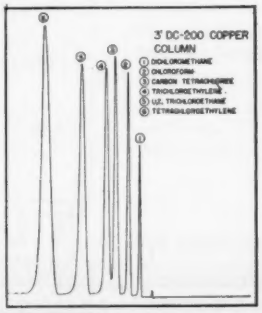

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Chemical Instrumentation

Most of the gases used as carriers are available in tanks in adequate purity for chromatographic purposes. In the case of certain of the detection systems, e.g., the flame ionization detector, the sensitivity is so great that it is sometimes desirable to submit the carrier gas to a rigorous purification step prior to introduction to the chromatograph. The Milton Roy Company, Philadelphia 18, Pennsylvania, offers a device that is useful for this purpose with hydrogen carriers. It is their Serfass Hydrogen Purifier, and consists of a bundle of heated palladium-silver alloy tubes. The palladium acts as a selective membrane, allowing only hydrogen to diffuse through, and holding back all impurity molecules. The principal parts of the unit are shown in Figure 2. Hydrogen flow rates as high as 200 cubic feet per hour are available.

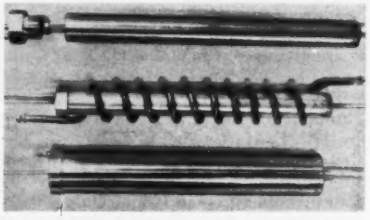

Figure 2. Principal parts of the Serfass Hydrogen Purifier. Top, a bundle of palladium-silver alloy tubes is encased in a stainless steel jacket. Middle, an electric heater is wound around the jacket. Bottom, the entire unit is encased in a heat insulated shield.

The chore of handling tanks of hydrogen can be avoided by the use of an electrolytic hydrogen generator, in which the gas is formed by the electrolysis of water. Such a device is made by Wilkens Instrument and Research Inc., Walnut Creek, California; it is the Aerograph Model 650 Hydrogen Generator ($225). Distilled water is electrolyzed in a polyethylene cell between stainless steel electrodes; the hydrogen gas so generated is filtered, and may be delivered to the gas chromatograph at flow rates from 7 to 40 ml/min.

For the purification of helium, passage through a packing of Linde Molecular Sieves cooled in dry ice or liquid air will remove all traces of contaminants down to the parts per billion range.

Flow Control

The rate of flow of the carrier gas through the chromatographic column is of fundamental importance in determining the performance of the chromatograph, and this flow rate should be accurately measured and closely controlled. The relation between HETP (height equivalent to a theoretical plate) of a column and the mean flow rate, $\bar{u}$, is expressed by the van Deemter equation:

$$\bar{H} = A + \frac{B}{\bar{u}} + \left[C_l + C_g\right] \bar{u}$$

where $\bar{H}$, the average HETP, is a measure

(*Continued on page A872*)

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of the extent of band broadening that occurs as a single component travels through the column; the A term arises from the non-uniformity of the column packing, and is often called the eddy effect contribution (in a capillary column there is no packing, and this term is zero); the $B/\bar{u}$ term arises from the diffusion occurring in the gas phase (B is proportional to the diffusion coefficient of the carrier gas; as the flow rate increases, the opportunity for random diffusion decreases, and $B/\bar{u}$ diminishes in magnitude, permitting sharper bands to be obtained); the $[C_l + C_g]\ \bar{u}$ term arises from the resistance to mass transfer between the liquid and gas phases (the more rapidly the gas molecules equilibrate at each point in the column between the liquid and gas phases, the smaller is $C_l + C_g$, and the sharper are the bands; the faster the molecules are swept through the column, the less opportunity is there for equilibration at each point, and the broader are the bands). It is evident that the second and third terms express trends in opposite senses, and hence there should be an optimum value of the flow rate at which these terms taken together contribute the minimum amount to the band broadening. The nature of these relationships is shown graphically in Figure 3.

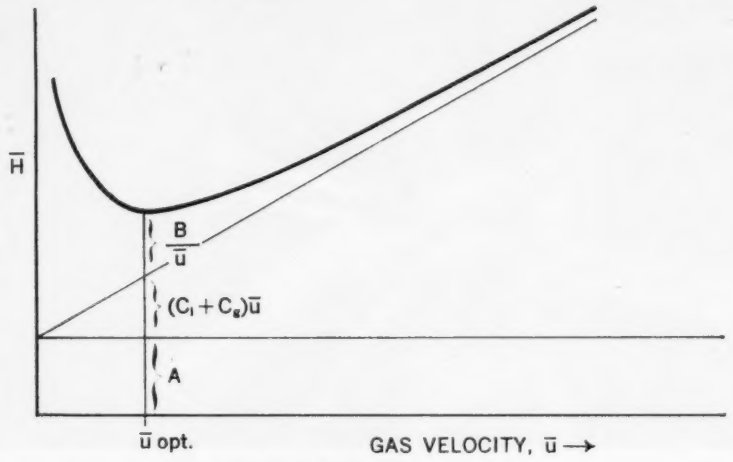


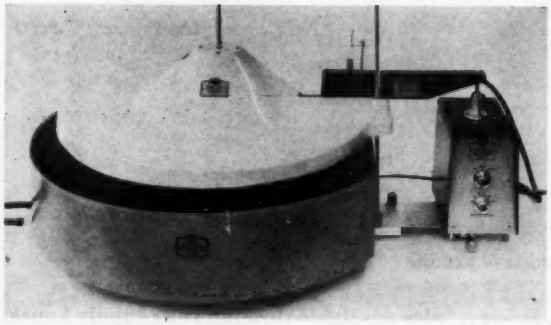
Figure 3. Graphical representation of the van Deemter equation, showing the relationship between $\bar{H}$, average HETP in the column, and the eddy diffusion (A), gas diffusion ($B/\bar{u}$), and mass transfer ($[C_l + C_g]\ \bar{u}$) terms. There is an optimum flow velocity at which the HETP is a minimum, i.e., the number of theoretical plates per unit column length is a maximum.

The carrier gas flow rates in gas chromatographs are generally measured by means of spherical ball float flowmeters, such as is illustrated in Figure 4. A precisely spherical ball is contained in a tube with an accurately uniform inner bore; as gas flows through the tube it raises the ball and causes it to float at such a level that the force of gravity acting down on the ball just balances the upward force exerted by the gas streaming around the ball and through the annular space between ball and wall. This height depends upon the gas velocity, but it also is a function of the density and viscosity of the gas, as well as the dimensions of the flowmeter ball and tube. Hence, a given flowmeter requires a set of calibrations; one for each different gas to be employed with it. The calibration is accurate only for the temperature and pressure at which it was determined, and only if the ball float and tube are scrupulously clean and dry.

Flowmeters of the type described above are available from most laboratory supply houses (e.g., Standard Scientific Supply Corp., New York 3, N. Y., has the Roger

(*Continued on page A874*)

NEW COOLING TROUGH for LKB AUTOMATIC RADIRAC FRACTION COLLECTOR



Where coldroom space is a problem and fraction collecting must be carried out, it is now possible to employ a new accessory from LKB that will allow test tubes to be kept at cold temperatures even though the entire apparatus is used outside of the coldroom.

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The LKB Drop Counter is shown positioned in one of the many operational possibilities for the LKB RadiRac Fraction Collector. The new LKB 3455A Cooling Trough and Dust Cover Assembly are also pictured.

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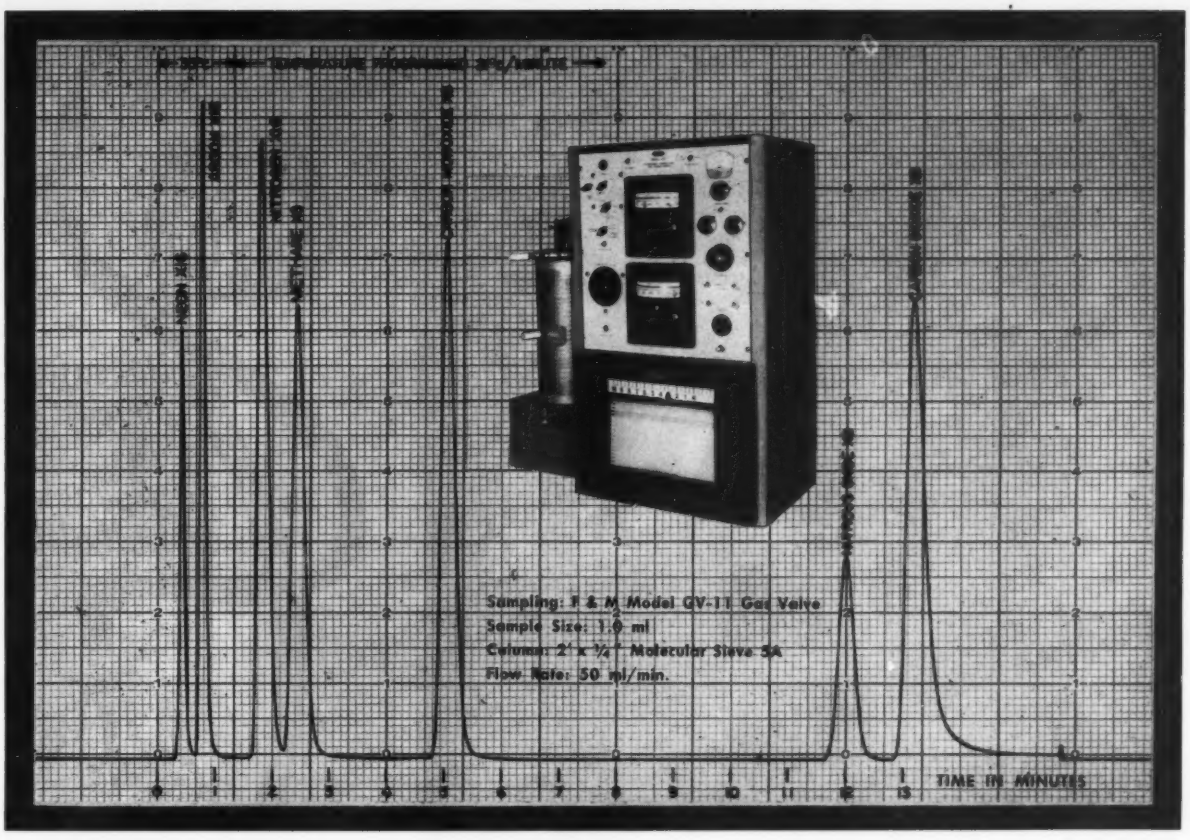

F & M Model 500 PROGRAMMED HIGH TEMPERATURE *Gas Chromatograph Separates 7 Inert Gases within* 15 *minutes*

Molecular sieve gas chromatographic columns are generally recognized for their capacity to separate fixed gases. Such columns, used in a well-designed, programmed temperature gas-chromatograph, provide the analyst with a versatile means of analyzing gas mixtures.

An example of this is shown in the above chromatogram. In less than 15 minutes, a complete separation was obtained for a mixture of neon, argon, nitrogen, methane, carbon monoxide, nitrous oxide, and carbon dioxide. Other gases for which molecular sieve columns are applicable include: ethane, ethylene, propane, hydrogen, deuterium, oxygen, nitric oxide, krypton, and xenon.

The above analysis was performed on an F & M Model 500 programmed temperature gas chromatograph, equipped with an F & M Model GV-11 gas sampling valve. This instrument combination provides the following advantages which are essential to reliable, accurate analyses of gas mixtures:

(1) *Repeatable Sampling.* Sample sizes are repeatable to within ± 0.25% with the Model GV-11 valve.

(2) *Precise Temperature Controls.* Temperatures of the column and detector are separately controlled from ambient-to 500°C by proportional controllers. The low thermal mass of the column oven makes possible a close adherence to a wide range of temperature programs.

(3) *Precise Flow Control.* The differential pressure flow control system used on the Model 500 assures a constant mass flow rate of carrier gas during a programmed temperature run.

Control of the variables discussed above is sufficiently good to give repeatability of ± 1% of both peak heights and retention times in temperature-programmed gas analyses.

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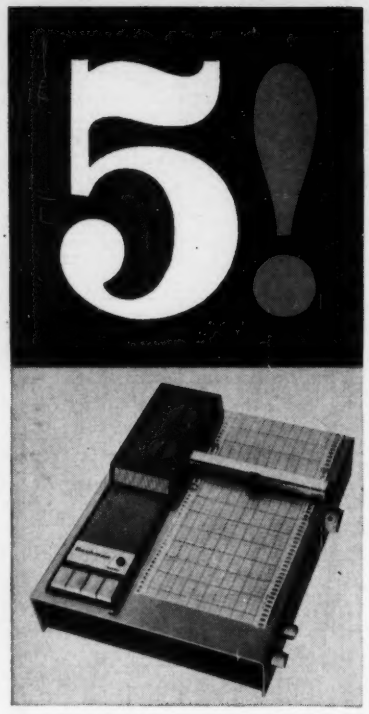

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Chemical Instrumentation

Gilmont Inc., line of flowmeters; Size No. 1, for 1 to 260 ml/min flow rates, $19.60). Ace Glass Inc., Vineland, New Jersey, has a line of flowmeters each of which contains two ball floats; one is of Pyrex glass, the other is stainless steel. The difference in weight is such that the lighter float reaches the top of the scale when the heavier one is one-third of the way up. Thus, the light float is useful for small flow rates, the heavy one for large flow rates, and the same flowmeter can cover an extended range (e.g., Size No. 1A, for 10 to 450 ml/min, $16.50).

Figure 4

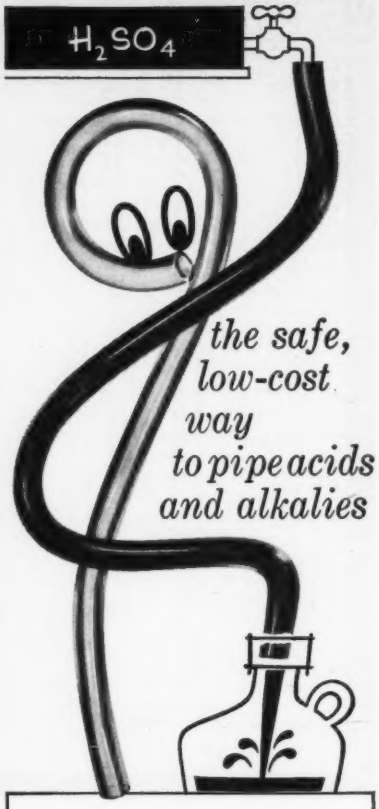
Figure 5

Figure 4. Ball float flowmeter. The gas stream to be measured flows through the precision bore tube and raises the spherical float to a height determined by the gas velocity.

Figure 5. Design of the Fischer and Porter Flowrator Meter, with Tri-Float metering tube. The flat surfaces provide guides to keep the float in proper position at all elevations.

The Fischer and Porter Co., Warminster, Pennsylvania, manufactures ball float flowmeters with a triangular cross-section, as shown in Figure 5. The three flat surfaces provide guides which keep the float centered at all points in the column (Model 448-209, with two interchangeable floats, sapphire for the range 7-1420 ml/min, and stainless steel for the range 15-2120 ml/min, $27).

Another approach to the measurement of the flow velocity of relatively slow-moving gas streams is the Pitot tube, one form of which is illustrated in Figure 6 (Model 5546, A. H. Thomas Co., $16.15). The gas flow is directed through a stopcock having a precisely defined orifice. The pressure difference generated across the orifice is a function of flow rate, and is measured by the U-tube manometer connected to the two sides of the stopcock. A constriction at the bottom of the ma-



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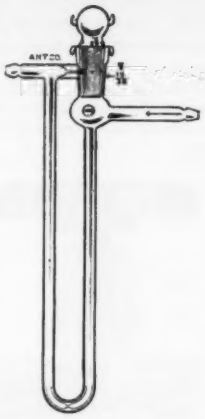


Figure 6. Pitot tube type of flowmeter. The U-tube constitutes a manometer that measures the pressure drop associated with the flow of gas through the stopcock orifice.

The soap bubble flow meter illustrated in Figure 7 is easily assembled from standard glassware, and is often used, especially for the calibration of other forms of flow meters. A soap bubble is caused to form at the base of a gas buret, and as the carrier gas flows into the buret from the exhaust of the gas chromatograph, the soap bubble rises. The volume through which the soap bubble moves per unit time gives the carrier gas flow rate.

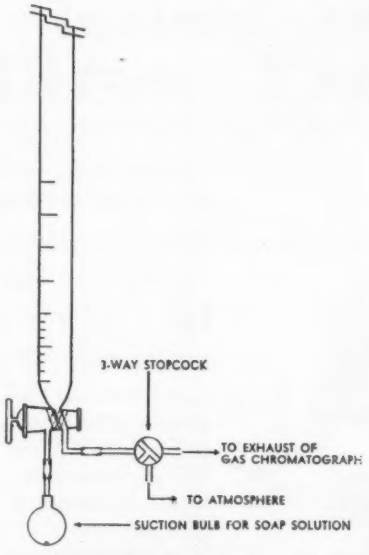
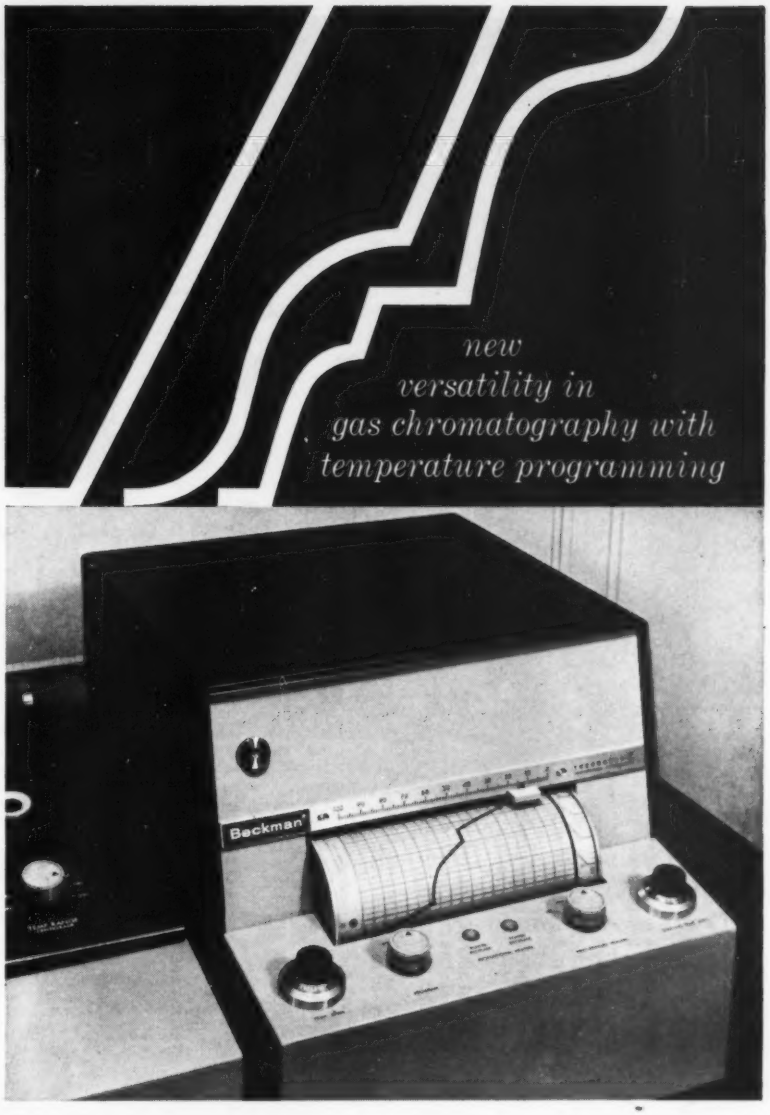


Figure 7. Principle of the design of the soap bubble flowmeter. A bubble formed near the base of the gas buret moves upward as carrier gas from the exhaust of the gas chromatograph enters, sweeping through a volume per unit time that can be read directly on the buret.

Other methods of flow measurement which are available, though not commonly employed in commercial gas chromatographs are: the wet-test meter (in which the gas is caused to bubble through a liquid; the bubbles cause a vane immersed

(*Continued on page A876*)



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in the liquid to rotate, producing an output deflection which can be read on a scale); the hot-wire anemometer (in which the temperature of a heated wire is influenced by the rate of flow of gas over its surface; the resistance of the wire is the measured parameter).

Figure 8. Design of a needle valve for control of gas flow. Rotation of handle moves the needle into or out of the gas stream, thus presenting a controllable restriction to the flow of gas.

Control of the flow rate of the carrier gas is generally accomplished with adequate precision by means of a conventional needle valve, such as that shown in Figure 8. The needle serves to provide a restriction to gas flow through the valve, and this restriction can be increased or decreased with good precision by screwing the needle into or out of the gas stream, respectively. The packing gasket or

Figure 9. Beckman Instruments liquid sampling syringe. ① plunger rod; ② Teflon plunger; ③, spacer; ④ adjusting screw; ⑦ split nylon cone; ⑨ collar stop; ⑩ setscrew.

gland is important, since it provides the seal that prevents gas from leaking out of

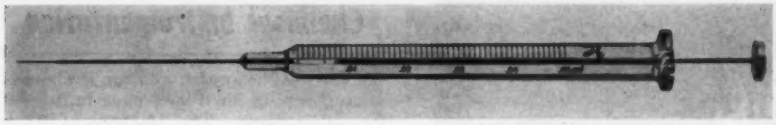

Figure 10. Hamilton gastight syringe for micro gas sampling.

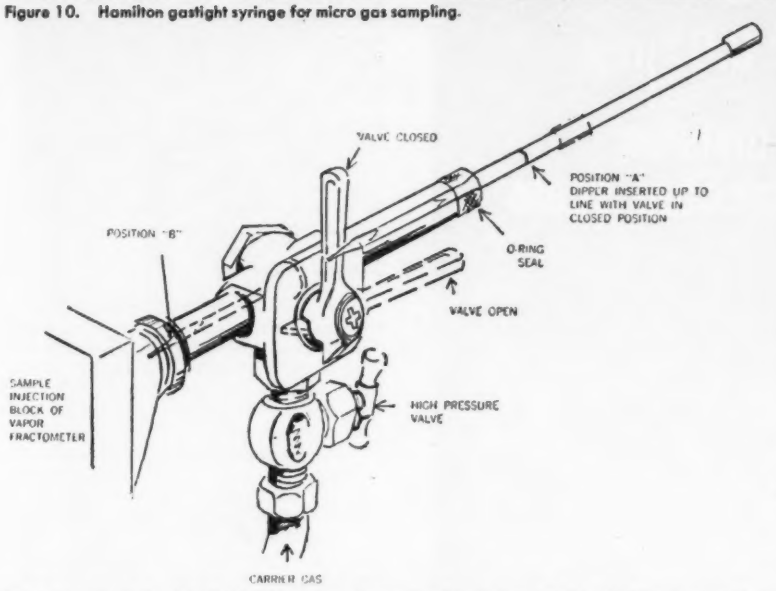

Figure 11. Principle of the Perkin-Elmer micro-dipper sampling system. The sketch shows two positions of the dipper in the sampling valve. In position "A," the dipper has been inserted through the O-ring seal. The valve handle is then turned to the open position, and carrier gas fills the chamber. The dipper is then pushed into position "B" until its dip contacts an orifice in the sample injection block. The high pressure valve is then opened, and the streaming of gas past the dipper tip causes the sample to be sucked out of the capillary and to be injected into the chromatograph.

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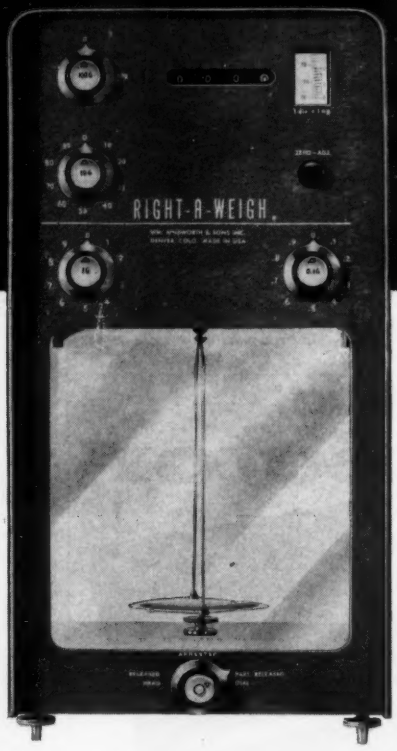

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the valve if the system pressure is above atmospheric, or air from leaking in if below atmospheric. The Matheson Company, East Rutherford, New Jersey, manufactures this type of valve with either Buna-N or Teflon packings, and brass, bronze, or stainless steel needles (e.g., No. 940SS, stainless steel and Teflon, $13.50). Other suppliers of gas flow control valves are : Ohio Chemical and Surgical Equipment Co., Madison 10, Wisconsin; Linde Air Products, Tonawanda, New York; Moore Products Co., Philadelphia 24, Pennsylvania.

Sample Injection

If the full potentialities of a gas chromatographic column are to be realized, it is essential that the sample injected be (*A*) sufficiently small so that it can be completely adsorbed by a very thin section of the column, and (*B*) injected all at once, so that a narrow "plug" of sample is inserted into the carrier gas stream, with no dilution of sample by carrier. A packed column, with thermal conductivity detector, normally operates at close to optimum resolution with liquid samples of 0.005 to 0.05 ml (5 to 50 microliters, or lambdas), and gas samples of 1 to 5 ml. For the liquid samples, it is desirable to measure and reproduce the sample size to ±1% or better. The syringe shown in Figure 9 has been developed by Beckman Instruments, Fullerton, California (price: $125), to permit delivery of samples in this volume range with a reproducibility of ±0.1% (i.e., $\pm 10^{-6}$ ml for a 10 lambda sample). It consists of a Teflon plunger which is driven a distance of 0.1 inch for every 10 lambda of liquid to be delivered, to a maximum of 50 lambda. The distance of travel is controlled by stops set with the use of accurately machined spacers. The diameter of the syringe needle is very small; total needle volume is only 0.004 ml.

The Hamilton Company, Whittier, California, makes a line of micro-syringes for gas samples. An example of one of their gas-tight syringes, with cemented-in needle, rhodium-plated stainless steel plunger, and Teflon gasket, is shown in Figure 10 (No. 1705N, $19). Micro-syringes for liquid samples are available with capacities as small as 1 lambda. Adapters are available for these syringes which serve to improve the reproducibility of volume delivery by increasing for the user the readibility of the distance traveled by the plunger.

Micro-syringes for liquid sampling are also available from Scientific Kit Company, Washington, Pennsylvania (Model 110, 10 lambda capacity, $9).

An alternative to the positive displacement syringe is the use of capillary action to cause a small pipet to fill with the liquid micro-sample, and the subsequent injection of the contents of the pipet into the gas chromatograph. This is the principle of the Perkin-Elmer capillary dipper system (complete with 4 dippers, $265), illustrated schematically in Figure 11. The capillary pipet, or dipper, is first filled with the liquid

(*Continued on page A881*)

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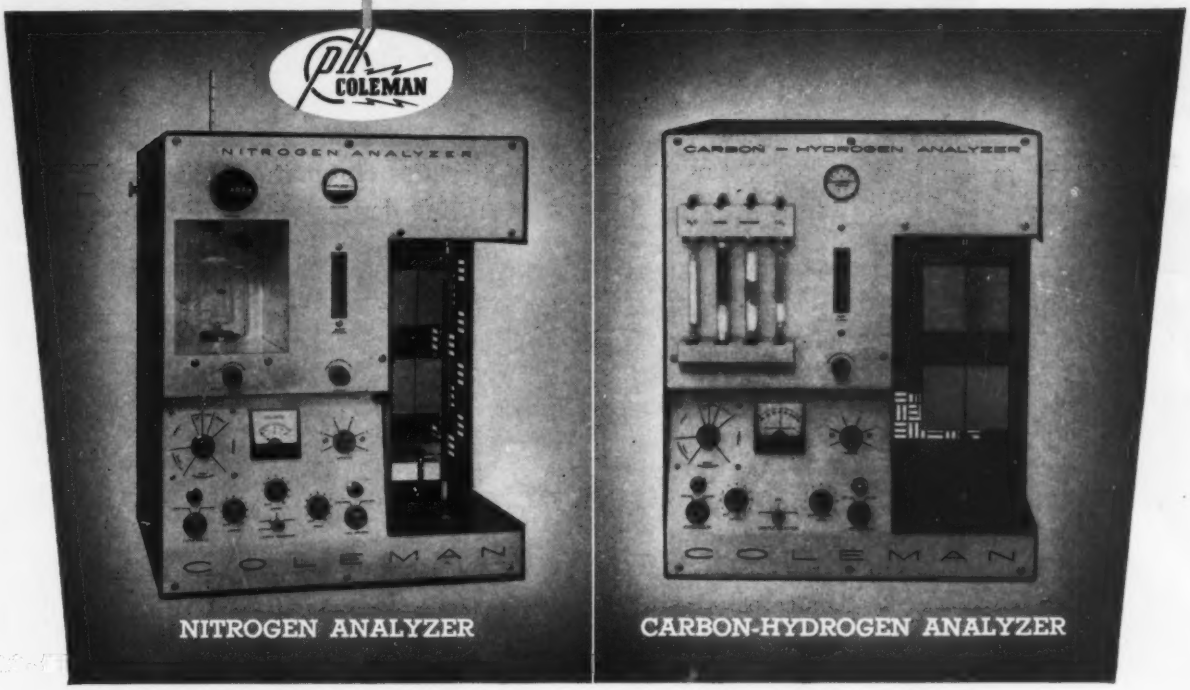

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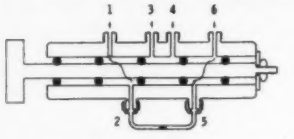
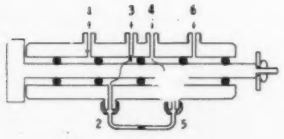
sample. It is then inserted part way into the sampling system, so that the barrel of the dipper seals the interior of the valve from the atmosphere. The chamber is flushed with carrier gas, and then the dipper is pushed further in, until it is close to the inlet orifice of the gas chromatograph. The carrier gas valve is then opened, and the streaming of this gas past the dipper tip creates an aspiration effect that sucks the liquid sample out and injects it into the chromatographic column.

Special gas handling valves have been designed to facilitate the collection of a precisely defined gas sample, followed by insertion of this sample as a plug into the stream of carrier gas. The operating principle of the Aerograph six-way gas sampling valve (Wilkens Instrument and Research, Inc., Walnut Creek, California, Model XA-203, $90) is illustrated in Figure 12. This is a two-position, linear-displacement valve. In one position, carrier gas flows through the valve and into the chromatograph, while a separate loop of tubing is filled with the gas sample.



NORMAL POSITION



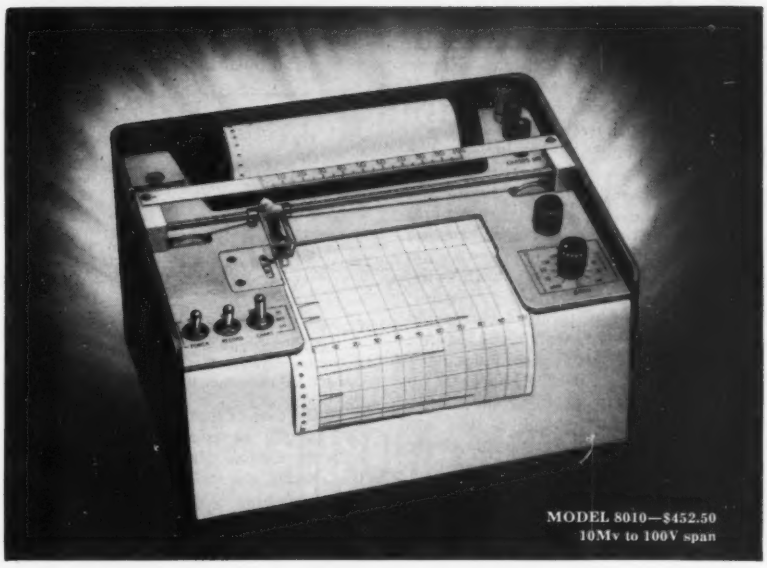
INJECTION POSITION

Figure 12. Diagrammatic representation of the functioning of the Aerograph six-way gas sampling valve. In the *normal position*, the plunger is retracted, and carrier gas flows from the tank into port 4, through the valve, out of port 3, and thence to the column. If the sample is introduced into port 6, it will fill the loop of tubing between 5 and 2, and emerge from port 1. If the plunger is now depressed, as in the lower diagram labeled *injection position*, the loop of tubing is inserted into the path of the carrier gas stream, and the sample is swept along with the carrier into the column.

The valve is then displaced to its other configuration, thereby causing the loop of sample gas to be inserted into the carrier gas line. A valve utilizing the same principle, but based upon a rotation of the valve to change from one configuration to the other is shown in Figure 13 (Beckman gas sampling valve, $210). A rotary valve based upon a similar design is available from Perkin-Elmer Corp., Norwalk, Connecticut (Gas sample valve assembly, $200); gas valves for chromatography are also made by Republic Manufacturing Co., Cleveland 35, Ohio; The Biotran Co., Houston 27, Texas; Julian H. Becker, Delft, Holland.

The sample handling capacity of a capillary (Golay) column is so small that

(*Continued on page A882*)



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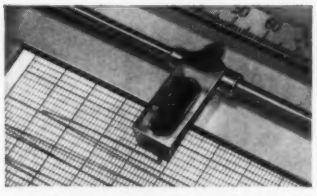

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even the micro sampling instrumentation described above is often not capable of delivering a small enough sample to the column. For such eventualities a stream-splitting technique is employed. The "plug" of sample gas injected into the carrier gas stream is caused to approach a tee-joint, where the gas flow splits into two streams; the principle of this scheme is illustrated in Figure 14. One stream goes to the capillary column (shown as the "capillary resistance" in the Figure); the other to a coil of tubing (represented as the "variable resistance" in the Figure). The ratio in which the gas divides itself between these two parallel paths is in inverse proportion to the resistances these paths offer to the passage of the gas through them. Thus, by suitable choice of the by-pass path dimensions, any desired fraction of the original sample can be introduced to the chromatographic column.

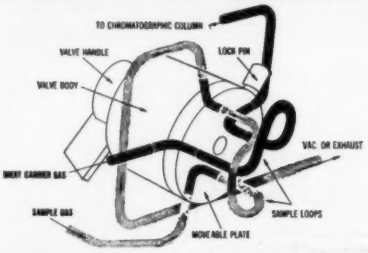

Figure 13. Phantom representation of principle of rotary gas sampling valve made by Beckman Instruments.

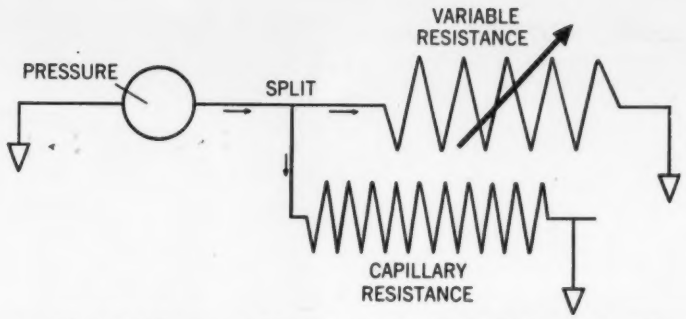

Figure 14. Schematic of the stream-splitting technique utilized to admit only a fraction of the sample to a capillary-type chromatographic column, in order to avoid overloading the column. The variable resistance shown in the diagram corresponds to a coil of tubing of lentgh and diameter appropriate to the desired resistance to gas flow.

In the case of liquids, it is essential that the sample be completely vaporized as soon as possible after being injected into the carrier gas stream, so that the narrowest possible "plug" of sample is formed. For this purpose, most gas chromatographs are provided with a preheated section of tubing, to bring the carrier gas up to column temperature before it reaches the place where the sample is injected, and a heated block is located at the injection port, in order that the liquid sample can be directed against a hot surface of high heat capacity.

Chromatographic Columns

The chromatographic distribution occurs in the column; hence, this component is the ultimate limiting factor in the performance of a gas chromatograph. Two principal classes of columns are now in use: (*A*) packed, and (*B*) capillary (Golay) columns.

A packed column consists of a straight length, U-tube, or coil of tubing that has been uniformly filled with a well-packed powder. The powder may be a dry, active adsorbent, such as the charcoal used in gas adsorption chromatography, or it may be an inert solid containing a film of a liquid, such as the crushed firebrick coated with any of a variety of non-volatile oils used in gas-liquid partition chromatography. A Golay column consists of a coil of capillary tubing, of 0.01 to 0.02 inches inner diameter, and containing 100 to 1000 or more feet of tubing; the inner walls of this capillary tubing are coated with a thin,

(*Continued on page A884*)

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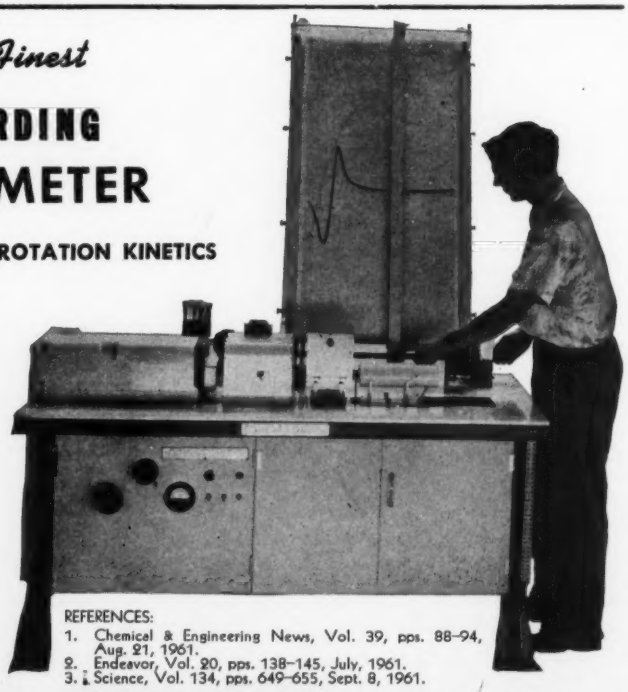

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2. Endeavor, Vol. 20, pps. 138–145, July, 1961.
3. Science, Vol. 134, pps. 649–655, Sept. 8, 1961.

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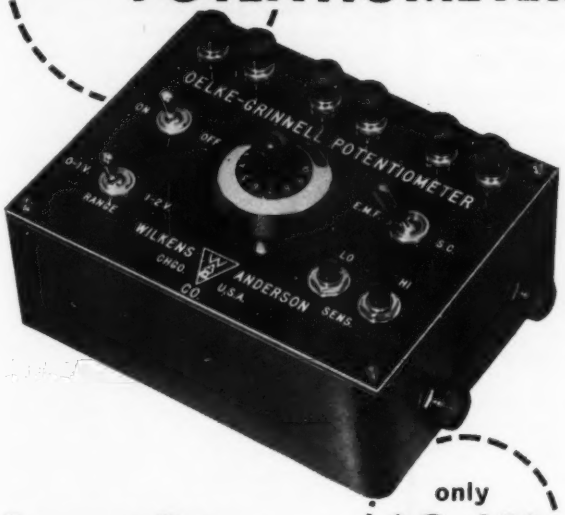

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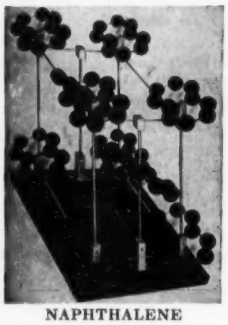

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Many gas chromatograph users make up their own columns because of the flexibility this permits in designing the column for the specific requirements of the chemical system to be separated, and for reasons of economy. Bulk quantities of column substrates and liquid phases are available from: Fisher Scientific Co., Pittsburgh 19, Pennsylvania; Analytical Engineering Laboratories, Hamden 18, Connecticut; Applied Science Laboratories, State College, Pennsylvania; and most of the chromatograph manufacturers. Tubing for columns is available from some of these sources, as well as from Superior Tube Co., Norristown, Pennsylvania; Corning Glass Works, Corning, New York (glass capillary columns).

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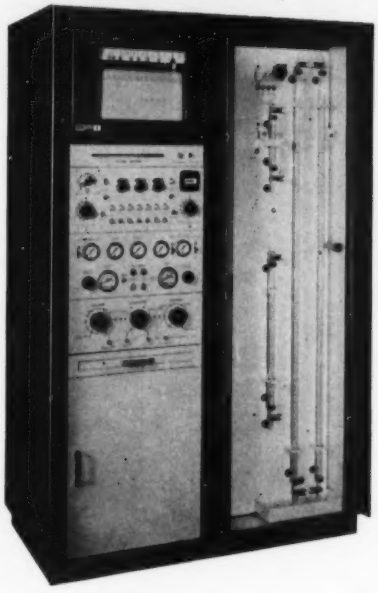

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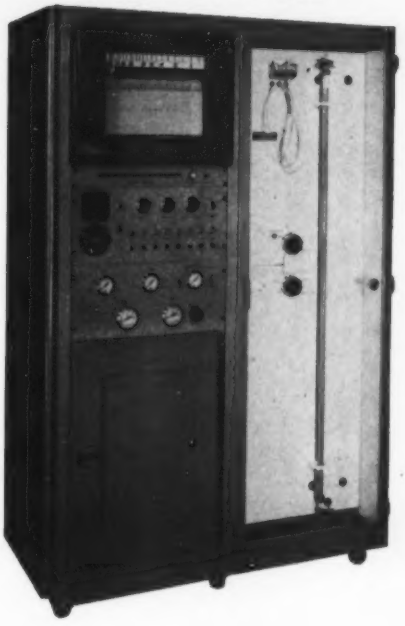

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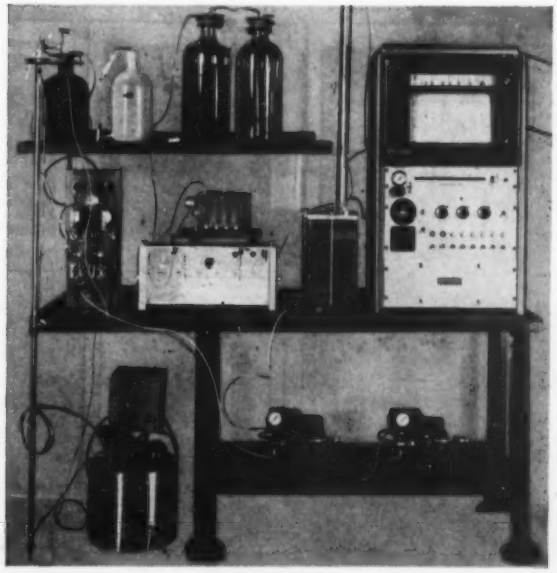

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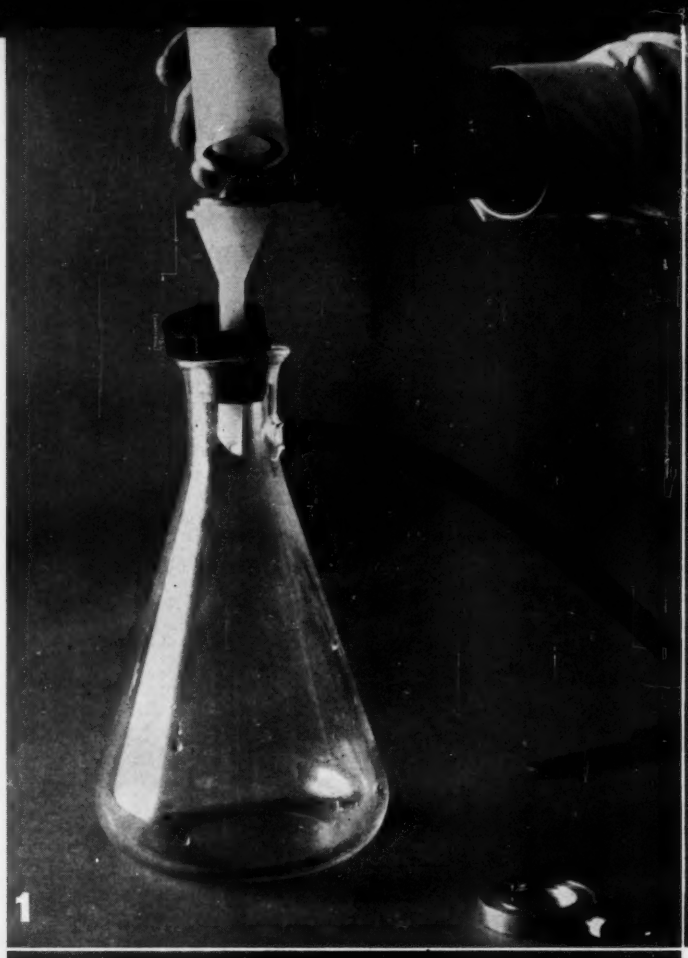

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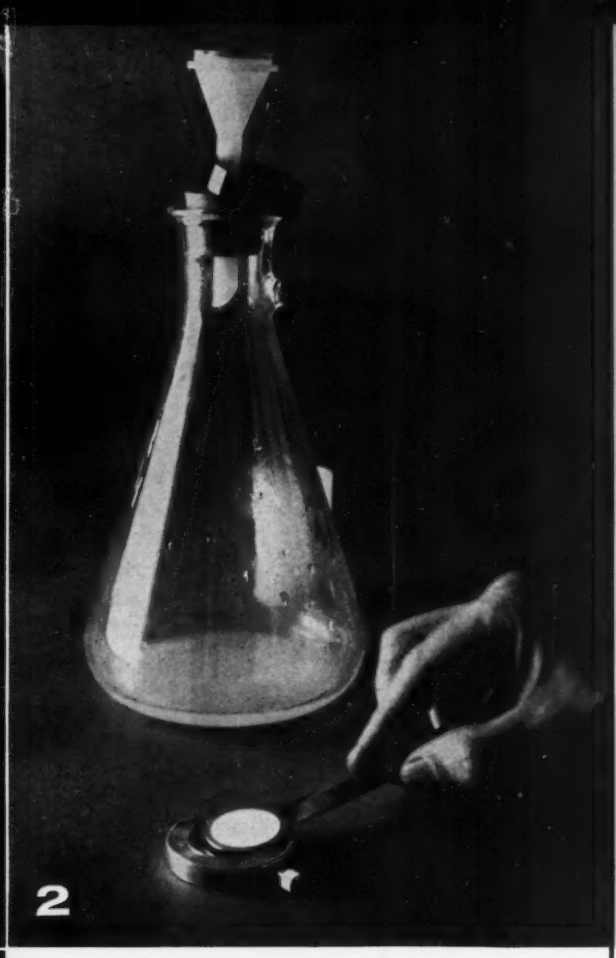

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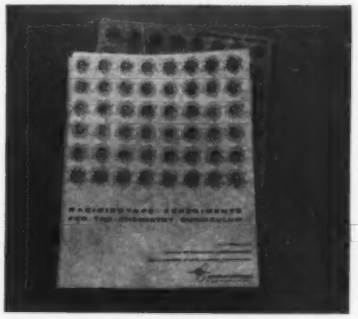

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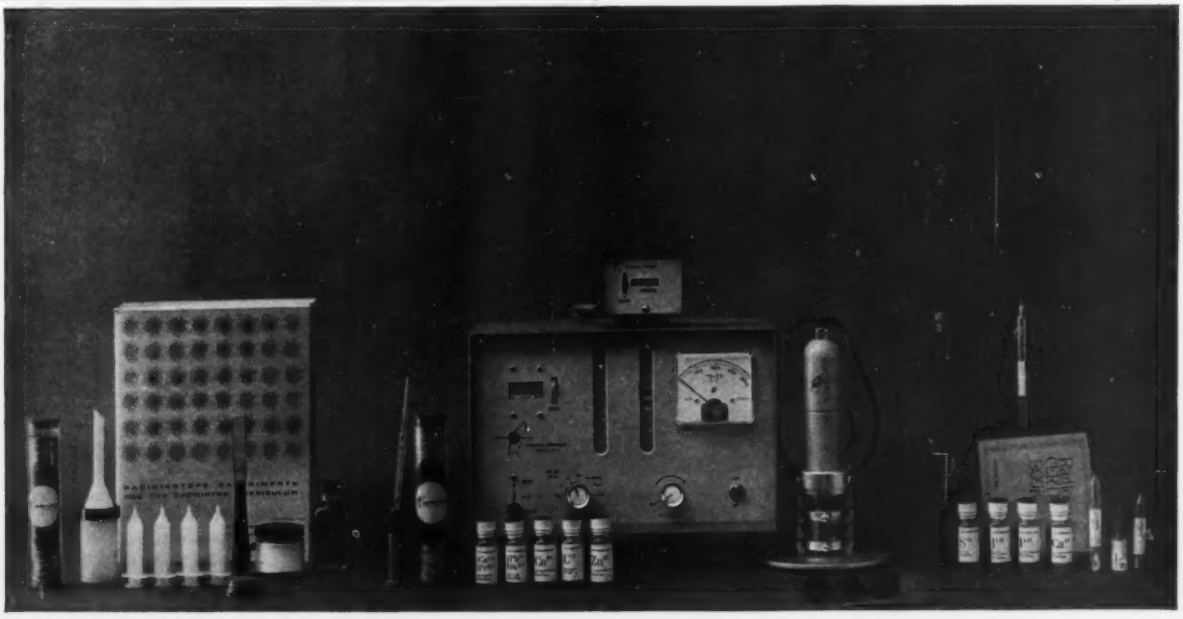

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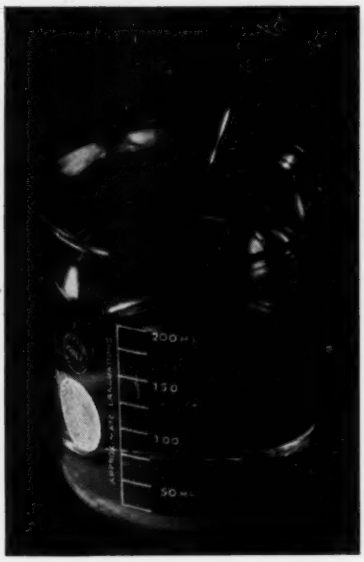

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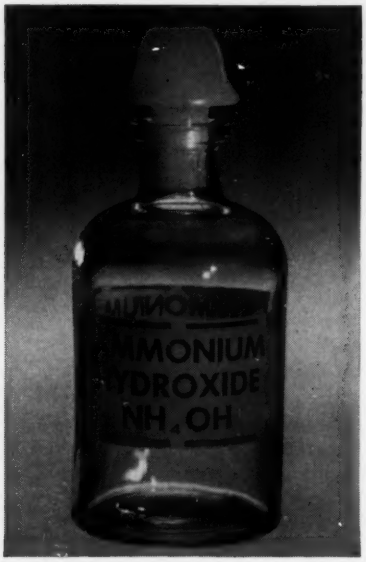

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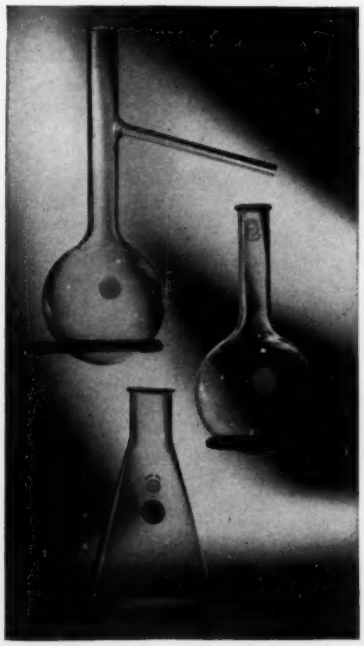

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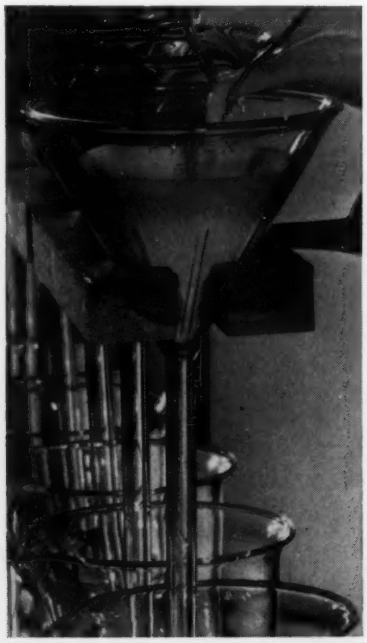

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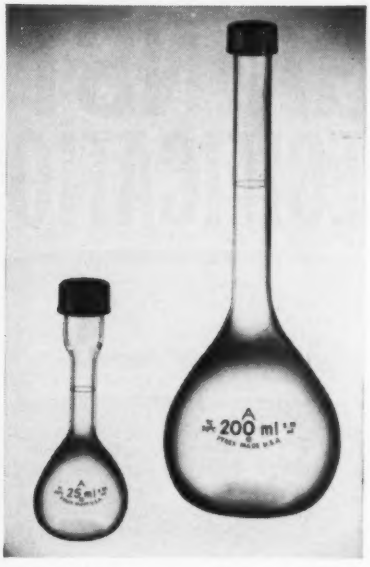

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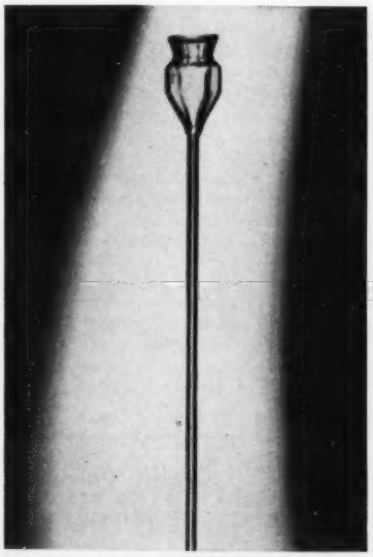

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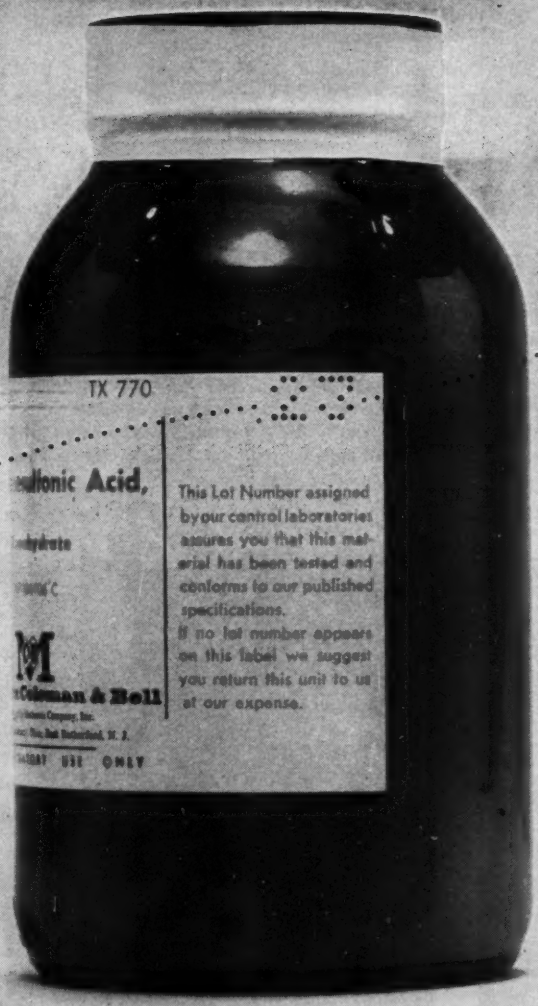
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CHEMICAL PROJECTS

Research Ideas for Young Chemists

Compiled by **JAY A. YOUNG,** Kings College, Wilkes-Barre, Penna.
JOHN K. TAYLOR, National Bureau of Standards, Washington, D. C.

Colloidal Gardens from Sodium Metasilicate

See DAMERELL, V. R., AND BROCK, H., *J. Chem. Educ.*, **26**, 148 (1949).

Colloidal, or chemical, gardens are familiar to almost every chemist and to many beginning students in chemistry. If a crystal of a soluble metal compound is placed on the bottom of a vessel filled with a solution of sodium silicate, a plant-like growth soon begins to form, and continues to grow, for several hours. The phenomenon is due to the formation of an insoluble metal silicate skin over the surface of the crystal of metal salt, which ruptures as liquid from the sodium silicate solution passes in, by osmosis, to the interior of the capsule surrounding the metal salt. On rupture, a new, enlarged sack consisting of insoluble metal silicate is again formed, and the process is repeated over and over again.

Damerell and Brock report that this growth is inhibited in solutions of sodium metasilicate, to which crystals of metallic chlorides have been added, when 16.5 ml of 3 *N* HCl is mixed with 330 ml of the sodium metasilicate solution before the metallic chloride crystals are added. Other concentrations of acid or base have a less inhibitory effect, or no effect at all. No explanation is offered.

Questions:

Verify the results reported by Damerell and Brock by repeating their work, by using solutions of sodium metasilicate of higher or lower concentration, and by using other soluble metal salts, in addition to the chlorides they used. Also, many different kinds of sodium silicate are available, with varying proportions of Na_2O and SiO_2. (In this sense, sodium metasilicate is said to be $1Na_2O:1SiO_2$.) Determine that the same inhibitory effect can or cannot be found for solutions of other sodium silicates containing varying amounts of added acid or base. Perhaps other acids, instead of HCl, will not show the same inhibitory effect. From the results of your investigation, suggest an explanation for the inhibitory effect described by Damerell and Brock, and test your explanation by further laboratory work.

The Growth of Lead Trees in Silicic Acid Gels

See HURD, C. B., AND LAMAREAUX, H. F., *J. Chem. Educ.*, **36**, 472-3 (1959).

Except at very low ionic concentrations, ions of a metal of low oxidation potential will be reduced by metals of higher oxidation potential and the less easily oxidized metal will precipitate out of solution. The oxidation and reduction half-reactions need not occur in the same place, provided that the electrons which are transferred can move from one location to the other. Hence, the less active metal often precipitates in dendritic forms, giving rise to the term "metallic tree." Lead trees form readily in gels, under almost any condition, but the conditions necessary for the formation of other metallic trees in gels are more critical. See TAFT, R., AND STAREK, J., *J. Chem. Educ.*, **7**, 1250 (1930) for comment on this point.

A silicic acid gel (other gels can also be used) containing dissolved lead ions is allowed to set. When a strip of zinc or other active metal is inserted into the gel, lead begins to precipitate almost at once and continues to precipitate for some time, forming a lead tree in the process. The conditions can be varied, with consequent changes, in some cases, in the character of the dendritic growths.

Questions:

Under what conditions will lead not form a dendritic precipitate in a gel? Explain your results and test your explanation by further laboratory work.

What conditions are required in order to form dendritic precipitates of one or more other reducible metals? Suggest reasons for these conditions and test your explanations by further laboratory work.

According to Hurd and Lamareaux, the dendrites formed by lead when different reducing agents are used, vary in appearance. Confirm this statement and suggest a reason for this phenomenon, testing your explanation by further work.

What other variables can you identify which will alter the nature of the lead dendrites which form? Explain these effects and verify your explanation.

The Use of Egg Albumin to Demonstrate Colloidal Phenomena

See KING, L. C., AND PETERSON, R. E., *J. Chem. Educ.*, **25**, 488 (1948).

The albumin particles in ordinary egg albumin are charged and hydrated. Simple tests, described in the article, have been designed to demonstrate these two properties.

Questions:

In what ways can the sign of the charge on the albumin particles be identified?

Show that when the *p*H of the albumin is lowered to a value less than the isoelectric *p*H of albumin, the sign of the charge on the albumin micellae is reversed. Explain this effect and devise tests to confirm your explanation.

How does the test described by King and Peterson demonstrate that the albumin micellae are hydrated? What other tests can you devise which indicate that the particles are hydrated?

Will albumin micellae solvate when placed in other than aqueous suspensions? Are the particles charged in a non-aqueous suspension? If so, can their sign be reversed?

What other sols exhibit some, or all, of the phenomena described by King and Peterson?

The Nylon Rope Trick

See MORGAN, P. W., AND KWOLEK, S. L., *J. Chem. Educ.*, **36**, 182–4 and 530 (1959).

Many different polyamides, to which class the Nylons belong, can be prepared by dissolving the two required reagents, a diamine and a diacid chloride, in water and in an organic solvent, respectively. The two solutions are placed in a beaker and, since they are immiscible, form two layers. A film of polymer may be produced at the interface between the two solutions. This film can be mechanically withdrawn from the beaker, forming a "rope" of polymer, which becomes longer and longer as the polymer is withdrawn, and new polymer forms at the interface, until, finally, the solutions are exhausted. The experiment is dramatic to watch and in addition presents an opportunity for many interesting studies.

Questions:

Prepare poly(hexamethylenesebacamide) and show that this substance is a polymer by applying the tests described in the article, and by applying other suitable tests.

Prepare one or more other polyamides and classify the differences in their properties on the basis of their different molecular structures.

Why is it necessary to use excess diamine, or additional base, in the aqueous solution? Test the validity of your answer to this question by further laboratory work.

Can other solvents, instead of water and carbon tetrachloride, be used to prepare a polyamide by interfacial polycondensation?

The authors state that when a hydrocarbon solvent is used in place of carbon tetrachloride, the polyamide which forms is sticky. Can you account for this result?

In *J. Chem. Educ.*, **26**, 611–2 (1949), BOSSERT, R. G., *et al.* describe an experimental study of the hydrolysis of Nylon. Using their procedure, or a suitable modification, can you separate the components of the poly(hexamethyleneadipamide) which is used in Nylon hose and then reconstitute this polymer using the procedure described by Morgan and Kwolek?

Devise a suitable procedure for the preparation of any polymer, other than a polyamide, by interfacial polycondensation.

Low Temperature Polymerization

See JENKINS, L. T., *J. Chem. Educ.*, **33**, 231 (1956).

Vinyl monomers—such as acrylonitrile, vinyl acetate, ethyl methacrylate, and others—can be polymerized in aqueous media by the addition of potassium persulfate followed by titanium(II) sulfate. The polymerization reaction is mildly exothermic; it can be initiated at room temperature. The potassium persulfate acts as an initiator and the titanium(II) sulfate as an activator. Hydroquinone will inhibit the polymerization.

Questions:

Will the polymerization reactions described by Jenkins take place in solvents other than water, such as ethyl alcohol, carbon tetrachloride, benzene, glacial acetic acid, or kerosene? Explain the results you obtain and test your explanation by further laboratory work.

Can other initiators, activators, and inhibitors, in addition to those mentioned above, be successfully used?

Is it true that the low temperature polymerization procedure described by Jenkins is only suitable for the polymerization of vinyl monomers? If so, can other low temperature procedures be found for the polymerization of other types of monomers?

Vinyl monomers are poisonous; some polymerization reactions are strongly exothermic. Do not inhale the vapors; quickly wash off any spills on your skin; use no more than test tube quantities.

Growth and Observation of Spherulites in Polyethylene

See BILLMEYER, F. W., JR., ET AL., *J. Chem. Educ.*, **37**, 460–1 (1960).

Thin films of molten (higher density) polyethylene form approximately spherical, partially-oriented aggregates of submicroscopic crystalline layers, called "spherulites," when they solidify by cooling. This property is characteristic of most crystalline high polymers. The size of the spherulites and other features of their appearance can be controlled by the experimenter. When viewed through a microscope between crossed Polaroids the effect is strikingly beautiful, and can be permanently recorded either with color or black and white film.

To control the thickness of the molten film, affix two small pieces of self-sticking transparent tape on a microscope slide, separated approximately 2 cm from each other. Draw the slide across the molten film so that the two pieces of tape rest on the glass surface on which the film has formed. The edge of the taped slide will be raised, by the thickness of the tape, above the film. This action will cause the film to spread, and its thickness will then be approximately equal to the thickness of the tape spacers. By using double or triple or more layers of tape, the thickness of the film can be adjusted to almost any desired value.

A suitable microscope eyepiece scale can be made by photographing a large, carefully drawn scale (white on black) on a blackboard. The developed film negative is cut to a suitable size and shape and inserted into the eyepiece of a microscope. It can be calibrated by measuring any suitably small object, of known size, using the markings on the scale.

Questions:

Is it true that, as stated in the article, the crystalline nuclei are progressively destroyed while the molten polymer is kept molten?

Show that the spacing of the rings of the spherulites depends upon the rate of cooling of the molten polymer. Suggest an explanation for this phenomenon and test your explanation by further laboratory work.

What other polymeric materials will form spherulites?

Does the presence of soluble impurities in the molten polymer affect the appearance of the solidified polymer, and can this effect be used to identify the dissolved impurity?

Will other substances, such as water, lubricating oil, anthracene, or sodium chloride, form spherulites or other interesting solid configurations when liquid films are cooled below their freezing point?

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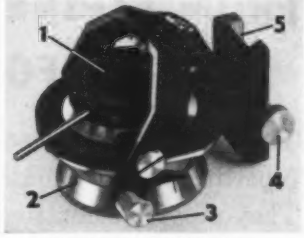

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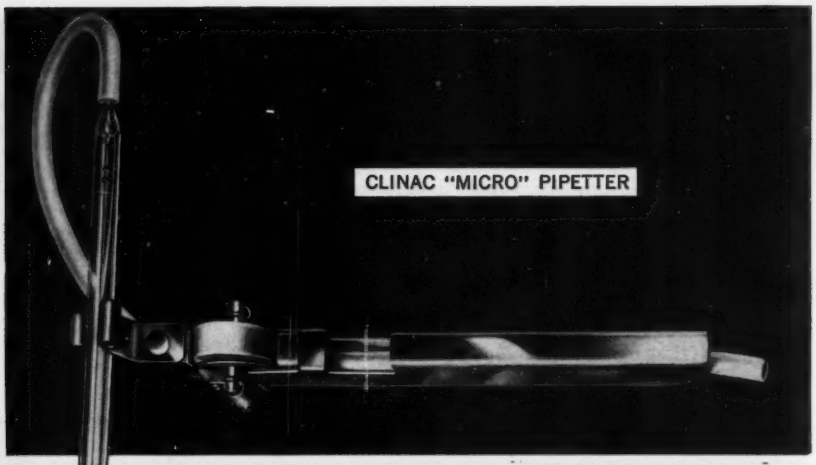

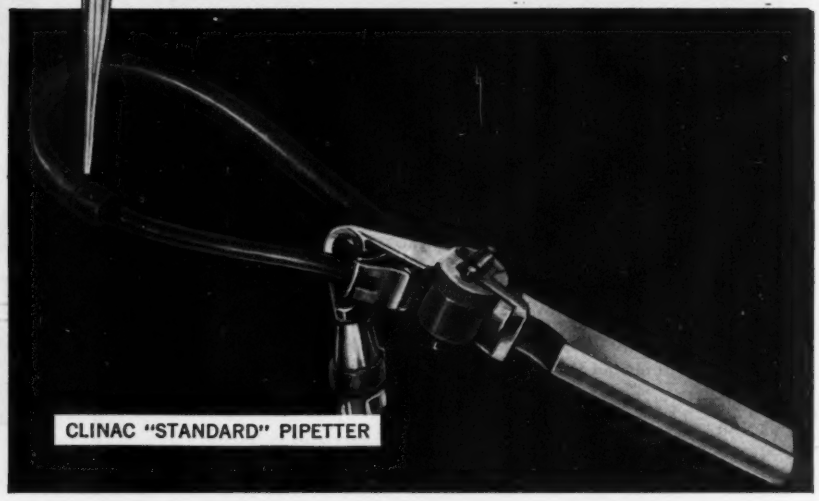

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FACTORS AFFECTING APPARENT KINDLING TEMPERATURE

A Chem Ed TESTED DEMONSTRATION

Submitted by: F. B. Dutton, Michigan State University
Checked by: Forty Michigan State University Traveling Science Teachers

EDITOR'S NOTE: *Innumerable variations of the following demonstration are frequently performed by teachers. It is suggested that these simple demonstrations contain more teaching opportunities than are commonly used.*

PREPARATION

Provide wood shavings, saw dust, and a short piece of a 2-in. × 4-in. block of wood; magnesium powder, ribbon, and a short length of 1-in. magnesium rod or similar mass; iron powder, steel wool, and an iron rod or bar; fresh calcium turnings, and a calcium turning that has been exposed to air for some time; matches, Bunsen burner, tongs, and asbestos or Transite sheet to protect table top.

DEMONSTRATION

1. Attempt to ignite 2 × 4 block with a match.
   Ignite shavings with a match.
   Gently blow or shake sawdust into the flame (hold the burner horizontally to prevent solids from falling into burner tube).
2. Hold magnesium block or bar in the flame.
   Ignite magnesium ribbon in the flame (use tongs).
   Sift magnesium powder into the flame (a salt shaker makes a good dispenser).
3. Repeat steps in (2) with iron bar, steel wool, and iron powder.
4. Attempt to ignite a calcium turning using tongs (a piece with clean surface can be ignited if the temperature can be raised to the kindling temperature rapidly).
   Repeat attempt with calcium turning which has been exposed to air and is well coated with oxide.

REMARKS

Assuming a constant air (oxygen) supply, consider the effects of the following: flame temperature, rate of production of heat by the flame, heat capacity of the object placed in the flame, heat conductivity of the object placed in the flame, temperature obtainable in the heated object, area and nature of surface of the object heated.

DETECTION OF WATER OF CRYSTALLIZATION—PREPARATION OF OXYGEN

A Chem Ed TESTED DEMONSTRATION

Submitted by: Puthiyaveetil Abdu Mohamed, The New College, Madras-14, India
Checked by: Leon E. Kauffman, Carlisle Senior High School, Carlisle, Pennsylvania

PREPARATION

Provide test tubes, a 100-ml flask, provision for collecting oxygen gas if desired, and fresh sodium peroxide; hydrated salts selected from the following list; and an anhydrous salt such as potassium sulfate.

$Al_2(SO_4)_3 \cdot 18H_2O$	$FeSO_4 \cdot 7H_2O$
$Fe_2(SO_4)_3 \cdot xH_2O$	$MnSO_4 \cdot 4H_2O$
$Na_2SO_4 \cdot 10H_2O$	$BaCl_2 \cdot 2H_2O$
$Na_2CO_3 \cdot 10H_2O$	$Na_2B_4O_7 \cdot 10H_2O$
$MgSO_4 \cdot 7H_2O$	$Na_3AsO_4 \cdot 12H_2O$
$CoSO_4 \cdot 7H_2O$	$Na_3PO_4 \cdot 12H_2O$
$NiSO_4 \cdot 7H_2O$	$CuSO_4 \cdot 5H_2O$
$ZnSO_4 \cdot 7H_2O$	

DEMONSTRATION

Place a small amount of the hydrate in a test tube and mix some fresh Na_2O_2 with the hydrate. Warm slightly and test for the evolution of oxygen. The test may be performed by inserting a glowing splinter into the test tube or, if one prefers, by passing the evolved gas into a solution of alkaline pyrogallol. Compare the results obtained with the hydrate with a similar experiment in which an anhydrous salt is used.

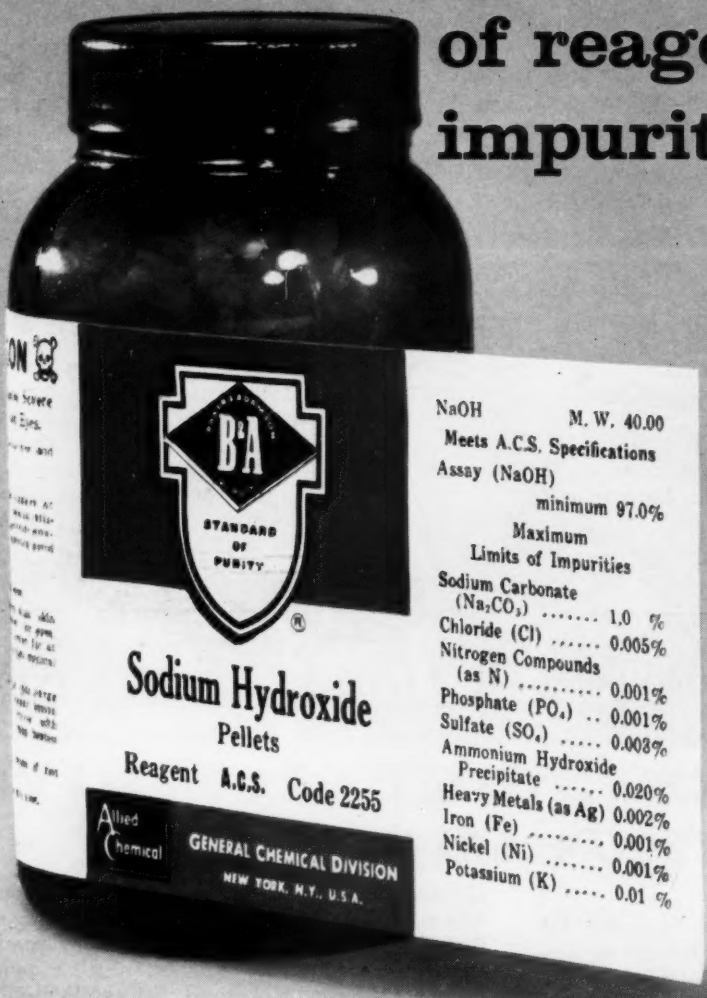
A considerable amount of oxygen may be generated by grinding together 2.5 grams of dry $MgSO_4 \cdot 7\ H_2O$ and 0.3 grams of dry $CoSO_4 \cdot H_2O$ (catalyst) and introducing 5 grams of fresh sodium peroxide in alternate layers in a dry 100 ml flask fitted with a delivery tube.

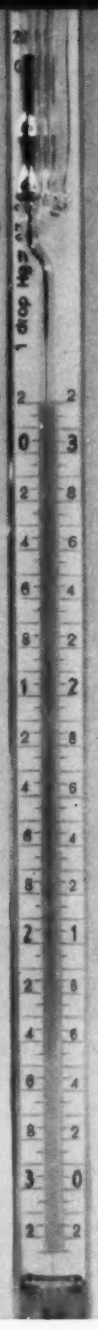
CAUTION

Highly deliquescent salts, salts of organic acids, and ammonium salts should not be included in these experiments. Even in the case of iron (II) salts which can (as reducing agents) consume some of the gas, considerable volumes of oxygen are generated.

DETACH PAGE BY CUTTING ALONG THIS LINE—INDIVIDUAL DEMONSTRATIONS MAY BE CUT APART AND PASTED ON 5 X 8 CARDS

What's your measure of reagent impurities?





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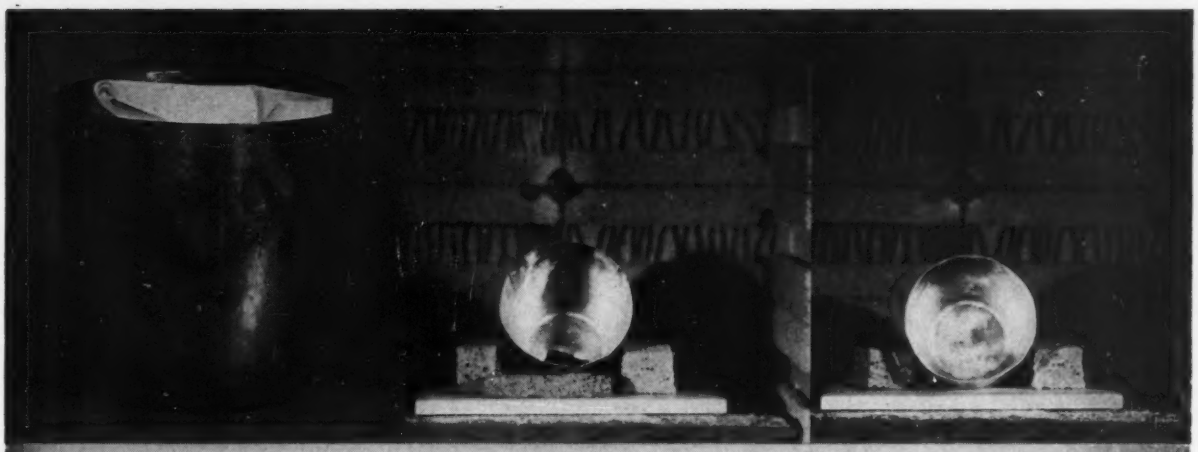

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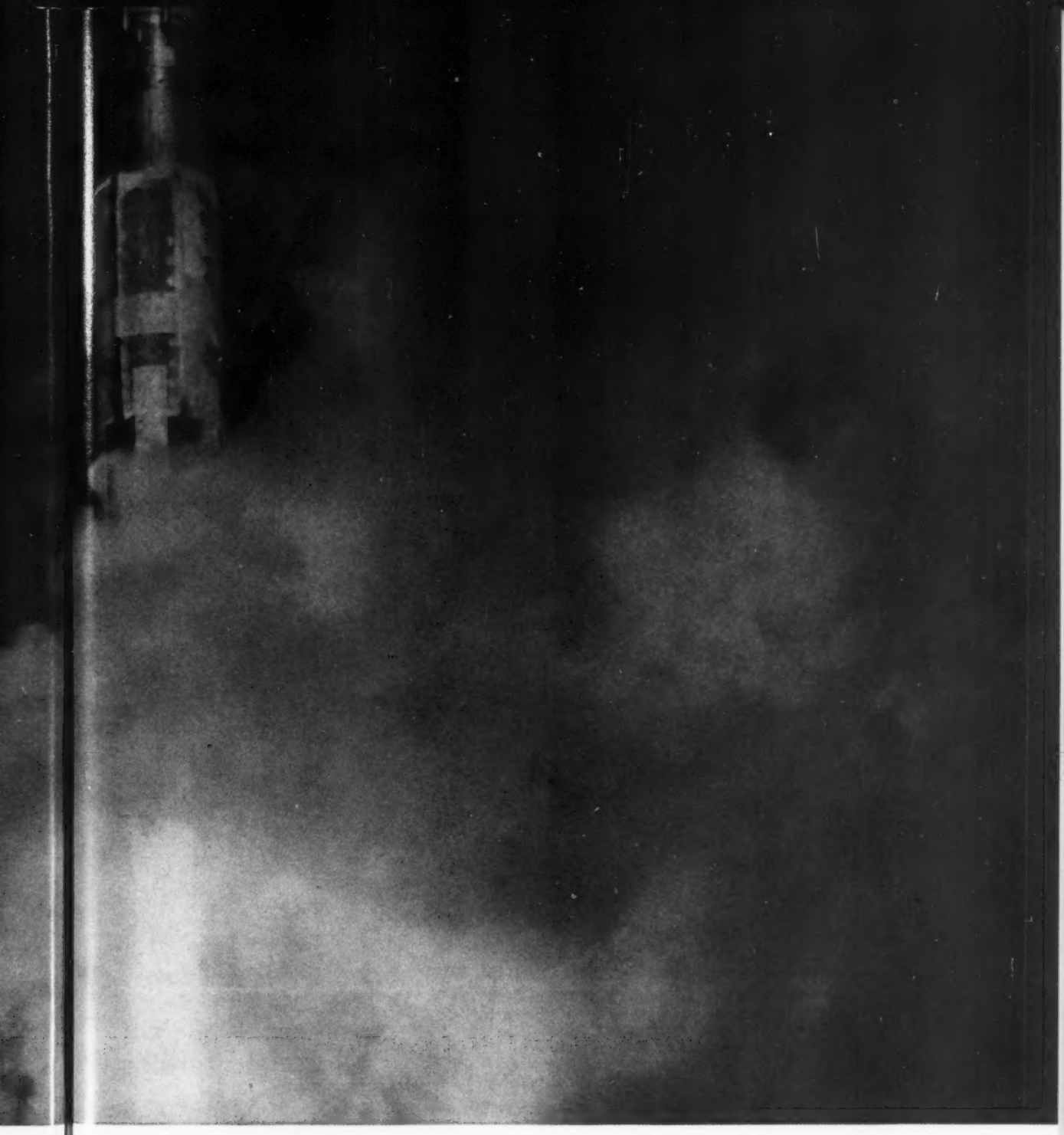

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PICKER NUCLEAR TRAINING INSTRUMENTS permit scheduling full laboratory courses in radioisotope techniques.

These transistorized instruments are remarkably compact (as you can see above). They are good-looking, rugged, easy to understand, and simple to use. They have the versatility and capacity not only for basic nuclear training, but for handling advanced techniques like pulse height anaylsis and rate function studies.

Their cost falls well within the reach of modest equipment budgets, such as those supported by A.E.C. grants-in-aid in pursuance of its Nuclear Education Program.* For details, please call any local Picker office (see 'phone book) or write Picker X-Ray Corporation, 25 South Broadway, White Plains, New York.

** The Picker Nuclear Division is prepared to help interested institutions in drawing up training programs in the use of nuclear techniques in biology, chemistry, medicine, agriculture, physics, and other fields.*

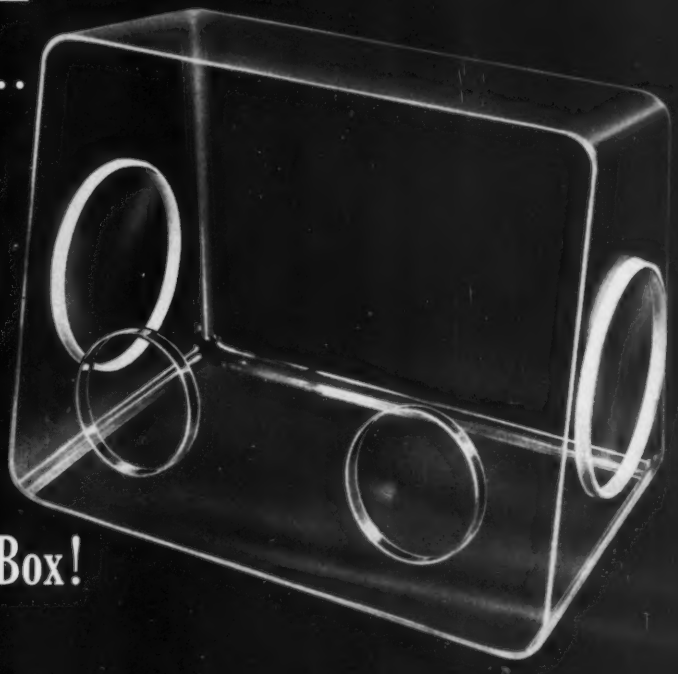
nuclear

Standing behind every Picker instrument is a local member of the Picker X-Ray national sales and service network.
He's there to protect your investment.
Because of him the user of a Picker instrument is never left stranded.

The features say –
$300 or $400 but...

$186

is the complete price of this Plexiglass Glove Box!



Don't let its economy fool you! This Manostat Plastic Glove Box boasts features that compare favorably with boxes costing twice the price!

■ 100% visibility through transparent 5/16" Plexiglass walls ■ Rounded corners for ease of cleaning and complete accessibility ■ Boxes may be interconnected with simple slip-on adapter to make multiple units ■ Easy access through 12-inch diameter side entry openings with readily removable covers ■ 8-inch circular flanged hand holes accept standard GB-1700 gloves ■ Adapts to so many available accessories for exceptional versatility... including new bellows gloves with replaceable hands, airlock, and coupling sleeves for joining two boxes. **GB-4000** Glove Box with two side access opening covers. . **$186.**

Write for Glove Box Bulletin

The
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E&G Co
26 N. Moore St., DPT. 422, N. Y. 13, N. Y.

A GOLDMINE of fine

The new Kimble catalog features full lines of KIMAX Beakers, Erlenmeyer Flasks, Petri Dishes, plus a host of money-saving innovations

KIMAX hard glass beakers, from 1 to 4000 ml . . .

KIMAX Erlenmeyer flasks through 6000 ml . . .

KIMAX Petri dishes, with covers and bottoms clearly distinguishable . . .

COLOR-CODED pipets . . . COLOR-CODED culture tubes . . . square flasks . . . round flasks . . . Raysorb low actinic ware . . .

these are but a few nuggets from the new Kimble Goldmine of fine laboratory glassware.

You'll find important improvements in hundreds of Kimble items in our new catalog. Take beakers, for instance. Made of tough KIMAX® hard glass. Beads are extra heavy. Glass is uniformly distributed throughout. Pour-out design minimizes dripping. Enameled marking area facilitates marking and erasures.

The entire Kimble line has been greatly expanded. Now, it's easy and profitable to buy the bulk of your needs from Kimble. Dealers in most states offer 28 per cent discount on orders of 150 cases or more.

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laboratory glassware

This new Kimble catalog should be on the shelf of every progressive laboratory. If you do not have one, mail the coupon today.

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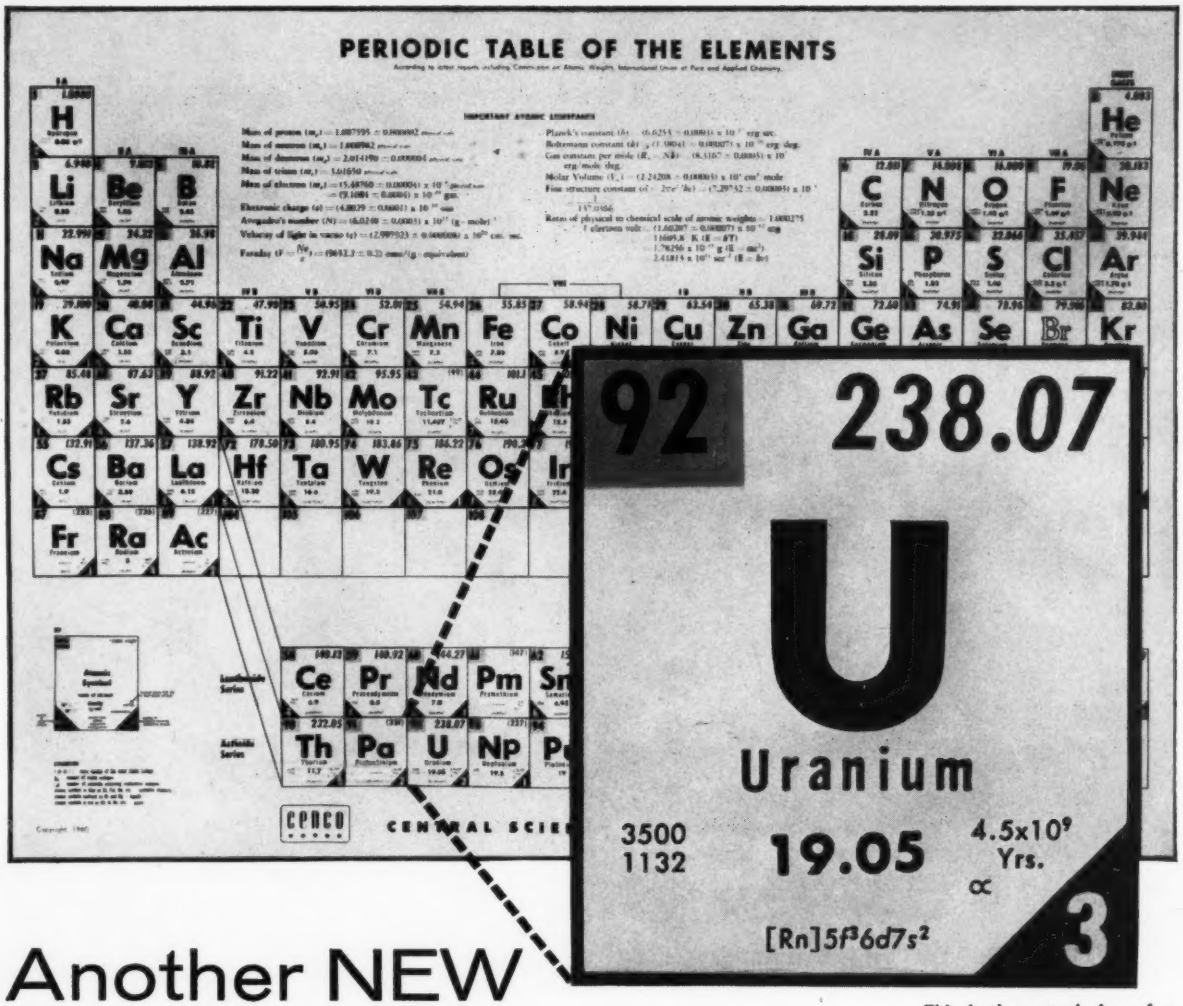

Another NEW
LECTURE ROOM
PERIODIC TABLE

This is the actual size of a single unit of this new chart.

Larger . . . Easy to read . . . Colorful . . . Includes atomic data

Here is the largest, most complete, up-to-the-minute Periodic Table now available. It includes all elements and number of naturally occurring radioactive and stable isotopes. Shows atomic number in large type as well as weight, density, boiling and melting points, electronic configuration, half-life, and other atomic constants for physics and chemistry.

This new, larger lecture room chart measures 62″ x 52″ and is reproduced in four colors on heavy stock. It is plastic coated for long life and easy cleaning.

No. 12056 Chart, fitted with wood slats at top and bottom as well as eyelets for hanging . . . . . each, **$7.50**

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ChemEd

Out of the EDITOR'S BASKET

Edited by William G. Kessel

When writing for additional information about new products or for new literature, your inquiry will receive prompt attention if you write on your firm or institution letterhead and mention the Journal of Chemical Education and the date of the particular issue. Pamphlets, booklets and similar literature are gratis unless otherwise specified.

New Apparatus and Equipment

▶ The first dual-channel liquid scintillation counting system designed for room temperature operation has been marketed by Nuclear-Chicago Corp. of 359 Howard Ave. at Nuclear Dr., Des Plaines, Illinois. The newly-developed instrument, called the Model 701, is designed for research applications requiring high sensitivity and efficiency in counting soft beta emitters such as carbon-14 or tritium. It is intended for laboratories interested in manually counting limited numbers of samples.

▶ A new X-ray analyzer crystal has been developed by the Isomet Corp. of Palisades Park, New Jersey. This crystal has a 2d spacing of 26A and extends the range of X-ray spectrometers by permitting the analysis of the lighter elements.

▶ A new, improved ball joint clamp has been introduced by the Manostat Corp., 26 N. Moore St., New York 13, N. Y. The new clamp permits users to exploit the significant potential advantages of ball joints to the fullest possible extent. This new concept in ball joint clamps provides a quick positive seal and instantaneous disconnect.

▶ Radiation Equipment & Accessories Corp., 665 Merrick Rd., Lynbrook, New York, now offers a selection of several inexpensive training systems for nuclear education. These systems include instruments using the same advanced transistor circuit techniques which are found in research equipment. These systems make it possible for all science courses to introduce the student to radioisotope techniques in his laboratory exercises. Complete sets of experiments utilizing the specific components of each R.E.A.C. training system are included.

▶ Four new "Thin Layer Evaporators" of JENA Glass Duran-50 (thermal expansion 32 × 10^{-7}), one of them to be used as "Molecular Distiller," have recently been developed by the JENAer Glaswerk Schott & Gen., Mainz West Germany, represented in the United States by Fish-Schurman Corp., 70 Portman Rd., New Rochelle, New York. At the present, detailed information is available, in German only, from the representatives.

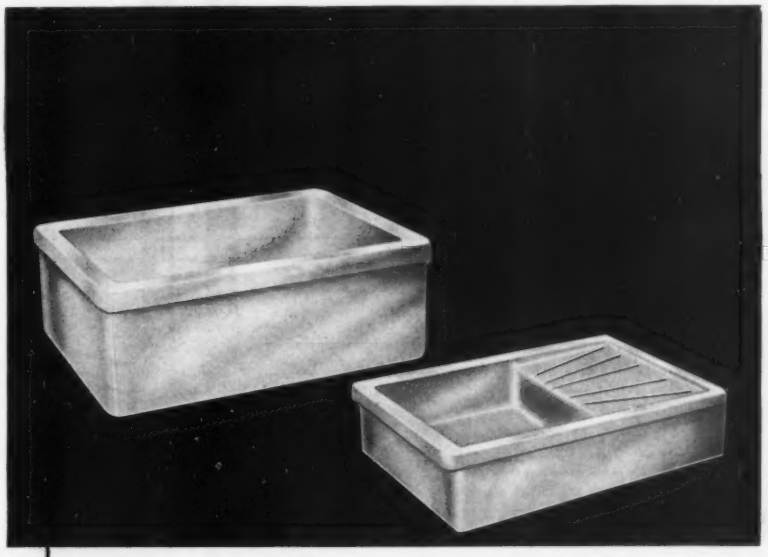
▶ The Vadosa, a unique new vacuum-producing apparatus which automatically draws up and measures any liquid in pipettes up to 25 ml., is now available for the first time to chemical, medical and biological laboratories in the United States from Hall Laboratory Products, 1267 Bushwick Ave., Brooklyn 7, New York.

▶ Scientific Industries, Inc., 220-05 97th Ave., Queens Village, New York, has made available a new multi-purpose rotator and they state that a wide range of laboratory mixing and agitation operations can now be handled with ease.

▶ Barnstead Still & Sterilizer Co., 168 Lanesville Terrace, Boston 31, Massachusetts, announces the development of automatic starting, stopping, and regenerating controls for its line of mixed-bed, and four-bed demineralizers in capacities up to 2500 gallons per hour.

▶ International Equipment Co., 1284 Soldiers Field Rd., Boston 35, Massachusetts, announces the development of the Maxiforce Ring, a new reinforcing ring for spinning 250 ml. plastic bottles at very high gravities.

(*Continued on page A906*)



See your Laboratory Furniture Dealer or write direct for a copy of Bulletin L-10.

This sink is solid chemical porcelain, as thoroughly corrosion-resistant as the porcelain crucibles and dishes the chemist uses . . . the only sink made today which possesses practically universal chemical resistance.

It is a beautiful sink. Trim in design, with smooth, rounded corners, inside and out. You may choose a mist-gray, surf-green, or sparkling white glaze — and the glaze is an integral part of the porcelain body — will never peel or flake off.

It possesses superb heat-shock resistance — and will withstand any heat shock encountered in laboratory usage.

Superior to stone, superior to porcelainized steel, superior to plastic, "U. S." chemical porcelain sinks are the first choice of laboratories everywhere.

90E-3

U. S. STONEWARE
AKRON 9, OHIO

Out of the EDITOR'S BASKET

▶ A new recrystallizer which automatically purifies small quantities (about one gram) of organic compounds in the laboratory with a minimum of attention by laboratory personnel has been introduced by Central Scientific Co., 1700 Irving Park Rd., Chicago 13, Illinois.

▶ Schuco Scientific, 75 Cliff St., New York 38, N. Y., is pleased to announce the "Pulverisette" line, said to be one of the most unique and versatile groups of laboratory mills and grinders on the market today. Manufactured by Alfred Fritsch Co., the "Pulverisette" incorporates many new developments in design and performance which make them truly outstanding units. An example of these fine instruments is the Pulverisette 2, a fully automatic universal laboratory mortar.

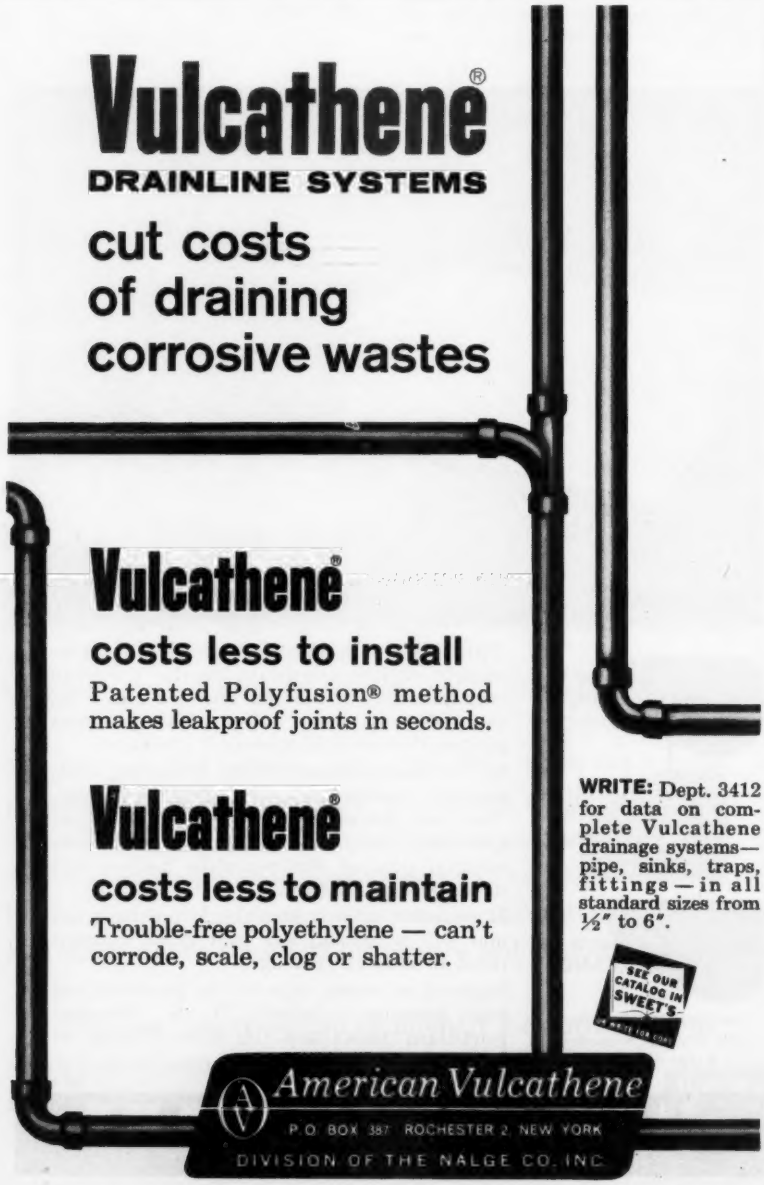
▶ A new glassworking hand tool, called the Spandle, is now available from Bethlehem Apparatus Co., Inc., Hellertown, Pennsylvania. The tool assists physicists, chemists, engineers and others to repair glass apparatus, to modify glass systems or to make simple apparatus. The principal advantage of the tool is that it enables a novice to make straight seals, ring seals, T's and Y's without arduous training and the skill required in professional glassblowing techniques. Chucking capacity of the Spandle ranges from 3 mm to 50 mm.

▶ A new universal positioning device for all-around laboratory use has been announced by Henry Troemner, Inc., 22nd and Master Sts., Philadelphia 21, Pennsylvania, manufacturers of precision balances and weights.

▶ Radiation Instrument Development Laboratory, Inc., 4501 W. North Ave., Melrose Park, Illinois recently announced the introduction of a Designer Series, claimed to be the most complete line of transistorized instruments ever presented to the nuclear field. Unlimited possibilities of combining instruments to design special systems are possible by the simple arrangement or rearrangement of plug-in instruments.

▶ Cahn Instrument Co., 14511 Paramount Blvd., Paramount, California, announces that their Gram electrobalance actually performs much better than they originally stated. Sensitivity (ultimate precision) is 0.1 microgram, instead of 1.0 microgram, and precision while taring is 10 parts per million of total load.

▶ Applied Research Associates, 403 E. 70th St., New York 21, N. Y., has developed a new high pressure ultrafiltration apparatus, the Araflo 65. This device permits rapid deproteination of plasma and the removal of colloids from colloidal solutions by high pressure filtration through supported cellophane or other membranes.



New Literature

● Write the Torsion Balance Co., Educational Aids Division, Clifton, New Jersey, for complete information on new Torbal educational aids for science students.

● A descriptive *Bulletin 794* on the new Beckman Solution Metering Pump is available from the Scientific and Process Instruments Division of Beckman Instruments, Inc., 2500 Harbor Blvd., Fullerton, California.

● Complete information and details on the Cenco-Lerner Lab-Jack are contained in a new six-page illustrated brochure just published by Central Scientific Co., 1700 Irving Park Rd., Chicago 13, Illinois.

● A list of references to papers on gas chromatography of steroids and related substances is included in the September issue of the *Gas-Chrom Newsletter*. This compilation includes work published up until June 1961 and may be obtained from Applied Science Laboratories, Inc., State College, Pennsylvania.

● *Selected Values of Chemical Thermodynamic Properties, Part I. Tables*, and *Part II. References*, by Frederick D. Rossini, Donald D. Wagman, William H. Evans, Samuel Levine, and Irving Jaffe. National Bureau of Standards Circular 500, Part I, reprinted July 1961, 828 pages, $2.50; Part II, reprinted July 1961, 481 pages, $2.25. (Order from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C.)

● A new catalogue showing complete specifications and listing on laboratory accessories has been published by Browne-Morse Co. of Muskegon, Michigan. It

is available from Department LA, to those persons interested in industrial, educational, medical or scientific laboratory applications.

● A new price list titled *Standard Carbon-14 and Tritium Compounds*, just issued by Volk Radiochemical Co., 8260 Elmwood Ave., Skokie, Illinois, briefs the chemical specifications, packaging, and pricing schedules for 139 ready-for-delivery isotope labeled materials.

● *Research Grants in Environmental Health in the Public Health Service* is the title of a booklet available from the U. S. Department of Health, Education and Welfare, Public Health Service, Bureau of State Services, Office of Resource Development, Washington 25, D. C.,

● *Catalogue 620* contains specifications and prices of a complete line of hydrometers and thermometers including general use from −200°C up to 1050°C, precision use up to $^1/_{100}$°C accuracy, ground joint, dial and special purpose types. Write Brooklyn Thermometer Co., Inc., 217–11 Merrick Blvd., Springfield Gardens 13, New York.

● High vacuum pumps and components are the subjects of a new six-page, illustrated brochure recently published by Central Scientific Co., 1700 W. Irving Park Rd., Chicago 13, Illinois.

● Over 100 different timers, from a basic stopwatch to a synchronous chronoscope for measuring intervals to $^1/_{100}$ of a second, for all segments of industry, are described in a new stopwatch selection guide issued by Heuer Timer Corp., 441 Lexington Ave., New York 17, N. Y.

● A 28-page brochure detailing the products and facilities of the company has just been released by Jefferson Chemical Co., Inc., P. O. Box 303, Houston 1, Texas. It provides technical data on all of the major product groupings in which the company is involved.

● ChemTrac Corp., 130 Alewife Brook Parkway, Cambridge, Massachusetts, has published a descriptive, 10 page catalogue, including price list, featuring its full line of Carbon-14 compounds and reference sources.

● The lastest catalogue *A-5* on *Atomic Instruments and Systems* is available from Baird-Atomic, Inc., 33 University Rd., Cambridge 38, Massachusetts.

● Cannon Instrument Co., P. O. Box 812, State College, Pennsylvania, announces the publication of *Viscometers Bulletin #20*, offering a complete line of all sizes and kinds of Viscometers.

● A new pocket-size bibliography, *Bulletin 795*, with more than 250 references on ultraviolet, visible, and near-infrared spectroscopy has been published by and is available from the Scientific and Process Instruments Division of Beckman Instruments, Inc., Fullerton, California.

● A new 20-page booklet on ethylene has

(*Continued on page A910*)

The Improved Welch

CATHETOMETER

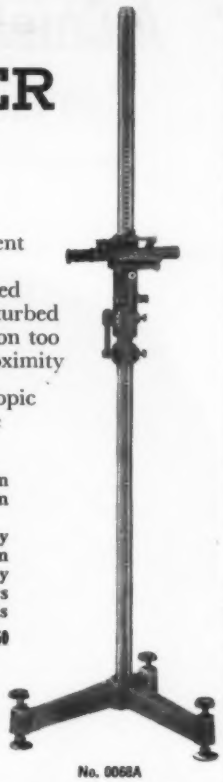
Significant recent engineering improvements in this instrument insure better performance than ever and more years of satisfactory service.

This cathetometer is designed for precise measurement of vertical distances through a range of 100 cm. It is particularly useful where the object to be observed is not conveniently accessible, or may easily be disturbed by physical contact, or must be located in a position too hazardous to permit the observer to be in close proximity

It is also commonly employed for qualitative telescopic viewing within the laboratory because of its versatile positioning adjustments and over-all stability.

Vertical Range	100 cm
Smallest Reading by Vernier	0.05 mm
Telescope	
Working Distance	45 cm to Infinity
Angular Magnification	12X at 45 cm 8X at Infinity
Reticle	90-degree crosshairs
Level Sensitivity	50 Seconds

No. 0068A—Each $257.50



THE WELCH SCIENTIFIC COMPANY
ESTABLISHED 1880
1515 Sedgwick St., Dept. D-1, CHICAGO 10, ILL., U.S.A.
Manufacturers of Scientific Instruments and Laboratory Apparatus

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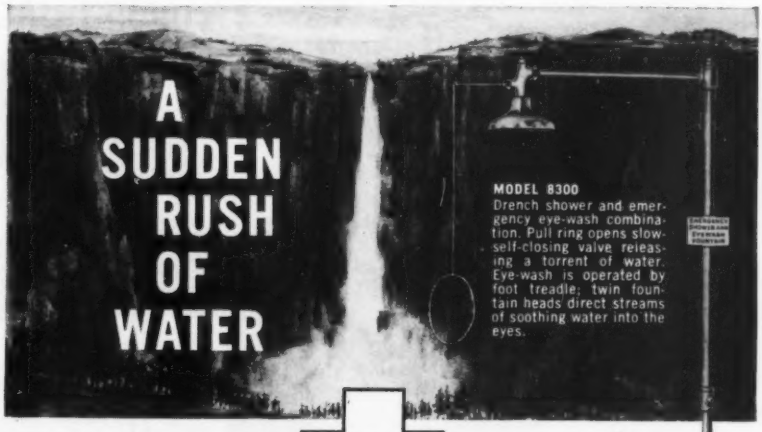
A SUDDEN RUSH OF WATER
MODEL 8300
Drench shower and emergency eye-wash combination. Pull ring opens slow-self-closing valve releasing a torrent of water. Eye-wash is operated by foot treadle; twin fountain heads direct streams of soothing water into the eyes.

for immediate **FIRST AID**
. . . in accidental body contamination

Injurious chemicals are instantly washed away by the cascading water from Haws Drench Showers. Simple pull-chain or foot treadle valves release a sudden rush of water—saving valuable seconds until medical help arrives. Chances of permanent injury are minimized, and insurance claims correspondingly lessened. HAWS Emergency Drench Showers are available in models to meet virtually any industrial need. WRITE TODAY—to find out the full story!

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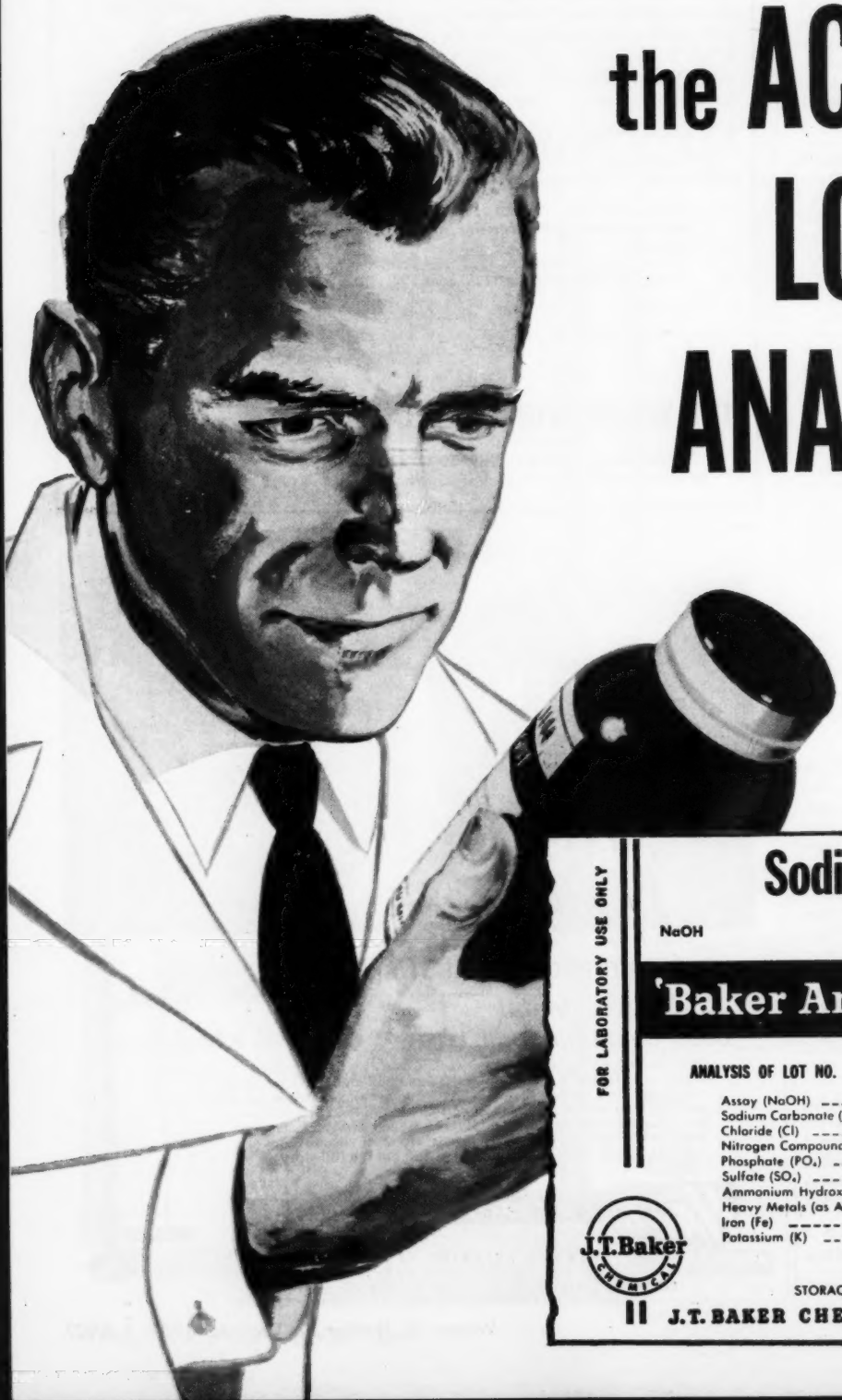
A Chief Chemist writes,

"We depend on, and use, the ACTUAL LOT ANALYSIS"



FOR LABORATORY USE ONLY

Sodium Hydroxide

50% SOLUTION

NaOH F. W. 39.9

'Baker Analyzed' *REAGENT*

ANALYSIS OF LOT NO. 23509

Assay (NaOH)	50.0	%
Sodium Carbonate (Na_2CO_3)	0.05	%
Chloride (Cl)	0.001	%
Nitrogen Compounds (as N)	0.0002	%
Phosphate (PO_4)	0.0002	%
Sulfate (SO_4)	0.0001	%
Ammonium Hydroxide Precipitate	0.005	%
Heavy Metals (as Ag)	0.0004	%
Iron (Fe)	0.0001	%
Potassium (K)	0.003	%

J.T.Baker CHEMICAL

STORAGE: Keep in tightly closed containers.

J.T. BAKER CHEMICAL CO., PHILLIPSBURG, N.J.

Read how chemists in major industries benefit from the more informative labeling on 'Baker Analyzed' Reagents

(Statements in response to a survey.)

FOOD Chemists

"We depend on, and use, the Actual Lot Analysis," writes the Chief Chemist of a major food company. *"In our work we must analyze for trace elements such as Fe, Pb, Zn and As. It's very important to know the amount of metal impurities in our reagents."*

"The Actual Lot Assay is of great importance to us in making standard solutions for our determinations."

Another food chemist states: *"The Actual Lot Analysis is very helpful in providing the nitrogen content of sodium hydroxide used for Kjeldahl determinations."* And from a third chemist: *"We use the Actual Lot Analysis to give an indication of the size of the reagent blank to be expected in trace mineral work."*

These men depend on, and use, the Actual Lot Analysis because they need precision laboratory tools. They can perform sensitive determinations with greater speed, accuracy and confidence.

PAINT Chemists

"We use the Actual Lot Analysis," writes a pigment chemist, *"as a check on our determinations of trace elements required to meet our specifications; for example, trace copper and manganese in rubber colors."*

"In trace analysis work," writes a second paint chemist, *"the Actual Lot Analysis of the reagents used is important. For example, when employing sodium peroxide as the oxidant in the determination of trace chlorine, the chloride impurity is important.*

'Baker Analyzed' Reagents are controlled to extremely low levels of impurity . . . the actual percentages found for key impurities are given on the label.

CLINICAL Chemists

"We depend on reagents with the Actual Lot Analysis," write two prominent clinical chemists.

From chemist #1: *"In bio-analytical procedures it's important for us to know the amount of impurities in our reagents."* And from chemist #2: *"We run thousands of determinations a year, especially for urea, sugars and carbon dioxide. It's very important, and saves us a lot of time, to know the amount of impurities in our required reagents."*

Both chemists are referring to the world-famous Actual Lot Analysis that appears on every 'Baker Analyzed' label. 'Baker Analyzed' Reagents offer the highest standards of purity and most informative labeling in the industry.

This is the label prepared for lot 23509 of 'Baker Analyzed' Reagent Sodium Hydroxide, 50% Solution. The **Actual Lot Analysis** provides the technologist with the exact level of critical impurities such as Nitrogen compounds and Carbonate content.

Every 'Baker Analyzed' Reagent is labeled with an **Actual Lot Analysis** that defines purity to the decimal, not just maximum limits. More than 300 are labeled with an **Actual Lot Assay,** not merely a range assay.

This is your LSR*. . .

an expert in laboratory supply who knows that *all* technologists benefit from the exact definition of purity provided by the 'Baker Analyzed' label. Your LSR represents one of the laboratory supply houses that stock J. T. Baker laboratory chemicals. His job is to help you get the chemicals, apparatus and glassware you need — when you need them. Call him — he wants to help you every way he can.

*Laboratory Supply Representative

J. T. Baker Chemical Co.

Phillipsburg, New Jersey

Out of the EDITOR'S BASKET

just been published by U. S. Industrial Chemicals Co., Division of National Distillers and Chemical Corp. This booklet is especially designed for developmental chemists and engineers using ethylene from cylinders. Copies are available from the Literature Dept., 99 Park Ave., New York 16, N. Y.

● University instructors, graduate and senior students will find great value in the technical paper *Experiments Using an Ionization Chamber and Vibrating Reed Electrometer for Radioactivity Measurements*, by Bert M. Tolbert, University of Colorado Department of Chemistry. It is available from Applied Physics Corp., 2724 S. Peck Rd., Monrovia, California.

● *Nickel Carbonyl. Part I.* A bibliography dealing with the preparation and properties of nickel carbonyl and covering the period 1890–1960. Bulletin ICB-25(I) Revision. Industrial Chemicals Section, The International Nickel Co., Inc., 67 Wall St., New York 5, N. Y.

● Analytical grade ion exchange resins (anion, cation, mixed bed, and special resins), "Cellex" ion exchange celluloses, and pre-calibrated resins, all specially prepared for use by research, analytical and pharmaceutical workers who are concerned with accurate, reproduceible ion exchange techniques free from resin-introduced impurities are described in the latest publication *Price List O* from Bio-Rad Laboratories, 32nd and Griffin Ave., Richmond, California.

New Chemicals

◇ HI-EFF-2, an ethylene glycol-succinic acid polyester, is the first in a new series of stationary phases for gas chromatography produced by Applied Science Laboratories, Inc., State College, Pennsylvania. Samples and descriptive literature are available.

◇ Kilocurie Cobalt-60 for teletherapy or non-destructive testing now available in international standard capsules, U. S. Nuclear all-welded Type 360, or in special capsules to suit customer's specifications. Normal focal spot is 1 cm for activities below 4000 curies. Detailed information on request from U. S. Nuclear Corp., Box 208, Burbank, California.

Miscellany

★ The *Dow Diamond*, Summer 1961, p. 18, features an article "In College Laboratories—An Emphasis on Safety." Harold W. Snyder, Safety Supervisor for the Dow Chemical Co., says safety training in college is important for many reasons. "It should be stressed early in the student's training as a measure to protect him and his fellow students. He will carry this training with him throughout his career just as he does other knowledge. Safety is as much a part of learning as formulas and techniques and should be an integral part of the curriculum."

★ "New Look in Museums," California's latest potent tool for public education is reported in *Vectors*, Volume III, No. 3, Third Quarter 1961, The Hughes Aircraft Co., publication. Don M. Muchmore, the Director of the California Museum of Science and Industry in Los Angeles, characterizes their approach by saying of the story telling technique, "We strive for a touch of Aristotle and a dash of Barnum."

★ Dr. Robert L. Pecsok, Professor of Chemistry, University of California, Los Angeles, will again be the Coordinator of a Gas Chromatography course, jointly sponsored by the Department of Chemistry, Physical Sciences Extension, and Engineering Extension, University of California, Los Angeles, to be conducted on the UCLA campus January 31—February 2, 1962.

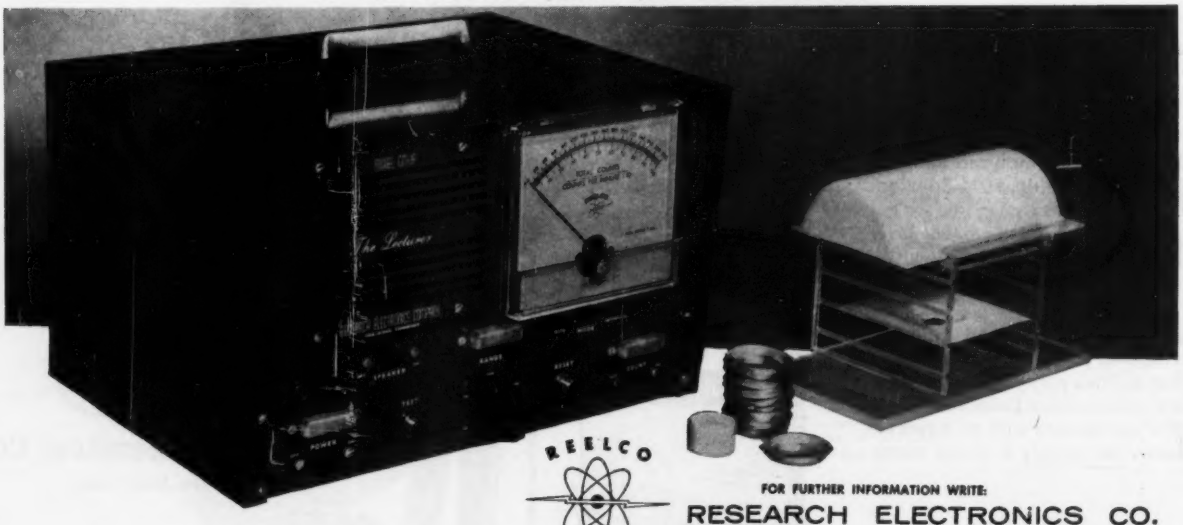
This intensive three day course is the fourth basic course presented in response to requests from industry.

The course is basic rather than advanced. It is aimed primarily to instruct personnel from industry, although it may be equally valuable to persons in academic and government laboratories.

The fee for the course will be $100.00; additional information and application blanks may be obtained by phoning or writing to H. L. Tallman, Physical Sciences Extension, Room 6532, Engineering Building, University of California, Los Angeles 24, California.

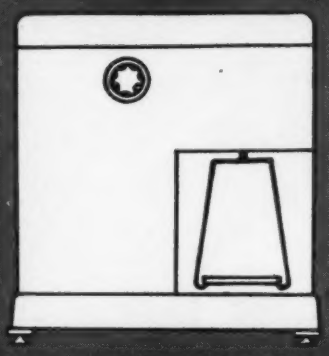
the all new REELCO quantitative GEIGER COUNTER

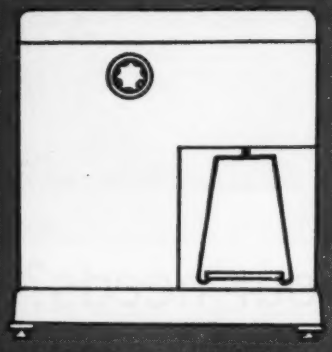
The "REELCO" Lecturer Model GM-1A Quantitative Geiger Counter, designed especially for science teachers by Oak Ridge National Laboratory, will generate new life and interest in the classroom. The "REELCO" Model GM-1A has many new superior features: **Large 4-inch Speaker-control panel switch—provides ample sound • for entire classroom • flashing indicator light—easily visible—Permits cutting of audio without sacrificing the effects of the demonstrators • Large 4½-inch, 2%, meter easily read—Highly accurate—Superior Visibility—Specifically designed for the classroom Provides count rate operation plus count accumulation.**



FOR FURTHER INFORMATION WRITE:
RESEARCH ELECTRONICS CO.
121 E. IRVING LANE OAK RIDGE, TENNESSEE

OUTWARD APPEARANCES ARE *Deceiving*





These two balances with the same outward appearance are fundamentally different. Both are single-pan, but one is a 2-knife-edge substitution balance, the other a 3-knife-edge, equal-arm balance.

This difference is of the greatest significance to instructors who use the balance to teach the principles of weighing or of measurement in general.

Teaching with a 3-knife-edge, single-pan balance, the instructor will explain that one major systematic error can be caused by inequality of the supposedly equal beam arms. As a corollary, he will show the students how the error can be checked quantitatively. Unfortunately, the construction of most 3-knife-edge, single-pan balances is such that this check is quite difficult to make for anyone but a trained balance technician. And transposition weighings, to eliminate the error, are not possible on these balances.

A system of measurement with a systematic error but no simple way to check the extent of the error is hardly one to use for teaching purposes

The 2-knife-edge, single-pan substitution balance compares object and weights on one and the same beam arm. It measures them with the same yardstick. A lever arm error cannot exist. This type of balance demonstrates to the student quite vividly how proper selection of the measuring method can reduce the number of systematic errors

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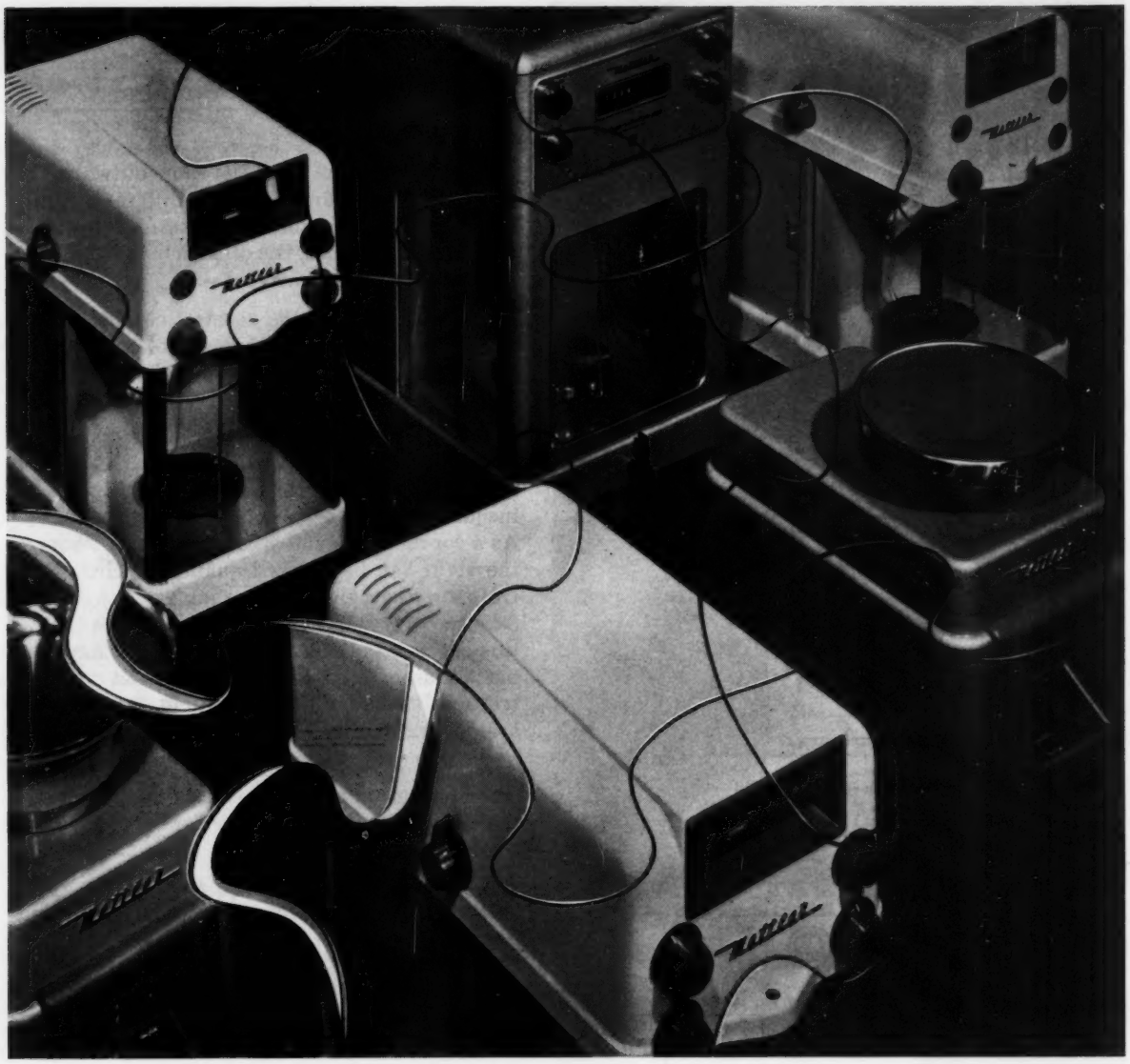

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Existence of a solvate between the butadiene monomer and the carbon-lithium bond of the "living" polymer is indicated by butadiene and styrene copolymerization experiments, conducted by Esso Research scientists, using butyllithium initiation.

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In other experiments with tetrahydrofuran, the copolymerization rate is increased, the reaction is of first order with respect to the ether, the styrene in the initial copolymer increases to 30%, and there is a shift in the structure of the polybutadiene segments from 1,4 to 1,2-enchainment.

A mechanistic model for this reaction has been developed. It suggests that the copolymer structure and the polymerization rate depend on the monomer concentration at the locus of polymerization. Butadiene appears to be 100 times more effective than styrene in the competition for the reactive site, perhaps due to the formation of a pseudo-6-membered-cyclic structure of low energy.

An ether may solvate lithium, making the C-Li bond more ionic, and thus increase polymerization rate. The stereochemistry would be altered by the solvation at the reaction site, increasing 1,2-enchainment and the availability of styrene. Solvates containing different amounts of ether may be formed and affect the 1,2-structure more than they affect initial copolymer composition.

These studies raise questions such as: can spectrophotometric evidence for the solvate be found? Can the temperature stability of such intermediates be determined? Will experiments with other monomers, ethers and solvents correlate with this mechanism?

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EDITORIALLY SPEAKING

Encouraging Creativity

There are two principal problems in the education of a chemist. One is to provide him with a reasonably full background of knowledge of previously established facts. These in essence are the vocabulary or working tools of his profession. The second is to stimulate to the fullest possible extent whatever creative ability or capacity for original thinking he may have.

The problem of teaching the factual information in a stimulating way, correlating the vast body of knowledge of chemistry with theory wherever possible and making the material interesting and stimulating rather than a dull recitation of facts, is a difficult one. Experienced teachers use many devices to achieve this end, recognized as desirable by all. I wish to deal primarily with the second objective of stimulating creativity.

Everyone who has answered a child's questions of "how" and "why" knows that the average intelligent child has a strong streak of curiosity. Our problem in education is to stimulate that curiosity, to help develop it into creativity, and to avoid dulling the student's mind by concentrating on solved problems and standard answers. There are mechanisms for doing this at every level of education. At the elementary and secondary school levels, students can be encouraged to undertake original projects, based upon whatever knowledge of the subject they have acquired at the time. Beginning in college, seminars dealing with current research and experimental problems to which the answers are unknown can be offered, and by the junior and senior year the student often has a sufficient background of knowledge to undertake an original experimental research problem.

By the graduate years, encouragement of creativity becomes the major problem in teaching at the highest level. In addition to encouraging the student to think for himself in his program of doctoral research, various devices that require the student to use his capacity for creativity can be incorporated in the curriculum. One that has been adopted by a number of universities is the substitution of the "proposition" for the oral or written examination at the highest level in the student's major field. Such a "proposition" by the student is the statement in writing of an original research problem suitable for experimental (or sometimes theoretical) study, with a review of the literature background and an outline of the method to be used to solve the problem. In defense of the"proposition" before a committee of the faculty, the student commonly is required to defend the originality, practicality, and significance of the problem that he proposes. This device has the advantage of requiring the student to think originally while he is studying for his highest level examinations, rather than simply trying to absorb the information in his major field so that he can reproduce or use it in his examinations. Long experience with the plan has shown that it is an excellent one for showing which students have developed farthest and have the greatest originality and scientific sophistication at the time that they present their "propositions." I believe that all students benefit greatly from this experience, and that it helps to instill good habits of reading the original literature, which the creative chemist must continue to do throughout all of his professional life.

All experienced teachers will be familiar with many devices for stimulating creativity; my object in these remarks is simply to call attention again to the importance of focusing a large part of the educational process on the stimulation of creativity. Most of us regard this as the chemist's most valuable asset.

ARTHUR C. COPE

ARTHUR C. COPE, 1961 President of the American Chemical Society, is a natural for that position. For years the Society has been calling on him to do battle with such crucial problems facing chemists and chemistry as publications policy and professional training standards. In spite of a busy life as an administrator, he manages to resonate between his office as head of the MIT Department of Chemistry and his organic research laboratory. His work in the latter won him the ACS Award in Pure Chemistry in 1944 and Columbia's Chandler Medal in 1958. A Hoosier by birth with collegiate training at Butler, he earned his doctorate at Wisconsin and held a postdoctoral fellowship at Harvard. He taught at Bryn Mawr and Columbia before assuming his present position at MIT. His comments here are an apt underscoring of his view on the real job of chemical education reported in *C and E News*, September 18, 1961.

E. Charles Evers
University of Pennsylvania
Philadelphia

The Nature of Metal-in-Amine Solutions

The reversible solubility of the alkali metals in anhydrous ammonia to produce blue to bronze colored solutions was first recorded by Weyl in 1864 (*64*). He inferred incorrectly that compounds like $NaNH_3$ were formed, which he termed "metal ammoniums." Although Seely as early as 1870 (*59*) advanced the opinion that "simple" solutions were obtained, his work seems to have been largely overlooked, and not until 1908 was significant progress made in establishing the nature of these systems. This came about primarily through the classical researches of C. A. Kraus (*39*) whose studies firmly established that true solutions were formed and that the negative carrier of electricity, the electron, was responsible for the color and transported a major fraction of the current. Furthermore it became apparent that dilute solutions might be described as "quasi-electrolytic" whereas the very concentrated solutions were essentially metallic.

Kraus proposed that metal in solution ionizes as a weak electrolyte, producing metal ions and solvated electrons in accord with the equation

$$xNH_3 + M = M^+ + e^-(NH_3)_x \qquad (1)$$

Ionization was assumed complete at infinite dilution and the equilibrium was presumed to shift to the left with increasing concentration. To explain the properties of more concentrated solutions it was suggested that solvated electrons were released to form "electrons essentially free in the metallic sense," i.e.

$$e^-(NH_3)_x = e^-(\text{free}) + xNH_3 \qquad (2)$$

The present concepts may be considered to be more sophisticated extensions of Kraus' theory, the principal modifications having been made to explain magnetic properties. However, no one model proposed to date adequately describes all the various phenomena, and there remains much to be done both in theory and in practice. It is becoming increasingly evident that, in order to advance our understanding of the nature of metal solutions, experiments must be devised which will provide us with a more detailed knowledge of the fine structure of these systems. To some extent applications of the newer techniques as electron spin and nuclear resonance measurements are providing information of this kind.

A number of excellent reviews have appeared during the past ten years (*3, 40, 65, 33, 63*); accordingly the more recent developments will be emphasized in this report, especially with regard to the choice of models for dilute solutions. Also we shall consider the manner in which certain properties vary as we proceed through the transition region from the dilute to the more concentrated metal-like state. In the transition region many of the properties and especially their derivatives undergo variations which demonstrate that significant changes are occurring in the structure of the system.

Dilute Solutions

Models

There is universal agreement that metal in solution dissociates, forming metal ions and electrons. The exact state of the electron, divorced from the metal ion, need not concern us for the moment. It will suffice to say that it is "solvated" and paramagnetic and, in ammonia at −34°, it has a conductance at infinite dilution of approximately 900 Kohlrausch units, corresponding to a mobility of roughly 0.01 $cm^2\,volt^{-1}\,sec^{-1}$.

There are essentially two schools of thought regarding the fate of the electron as the metal concentration is increased.

Cavitation Model (*52, 53, 25, 49, 36*). The cavitation model proposes that electrons, present singly in cavities created in the solvent, pair up with spins opposed as the concentration is increased, in accord with equilibrium

$$e^-(\text{am}) \rightleftharpoons {}^1/_2\, e_2^{-2}(\text{am}) \qquad (3)$$

Paramagnetic species are thus converted to diamagnetic pairs.

Expanded Metal Model (hereafter referred to as the B.L.A. model, named for the authors who proposed it) (*1*). The expanded metal concept differs from the cavitation model in that the solvated metal ion, M^+ (am), becomes an important entity in determining the state of the system. With increasing concentration there is association to monomer units, M (am), in the sense of the equilibrium

$$M^+(\text{am}) + e^-(\text{am}) \rightleftharpoons M(\text{am}) \qquad (4)$$

where the electron is assumed to circulate among the hydrogens of the ammonia molecules coordinated to the metal ion. Monomers then dimerize to the diamagnetic pair, M_2 (am), namely

$$M(\text{am}) \rightleftharpoons {}^1/_2\, M_2(\text{am}) \qquad (5)$$

which may be regarded as a pseudo hydrogen molecule, the species M being held together chiefly by exchange forces.

While a detailed description of the structure of these species is of prime theoretical interest and importance, it is not essential, *a priori*, that we have this information

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to test the applicability of these *concepts* in explaining the effect of temperature and concentration on phenomena such as conductance, magnetic susceptibility, and certain features of the absorption spectrum. The models themselves represent merely highly oversimplified means of describing the strong interactions which must exist among ions, electrons, and solvent molecules in a dielectric medium such as ammonia.

It should be pointed out that while most of our quantitative information has been obtained in ammonia, metal solutions may be prepared in a variety of solvents (*33*). A satisfactory model should therefore be capable of explaining data obtained in these solvents as well as in ammonia; in fact, studies in other solvents have been used successfully in correlating certain features of optical and magnetic behavior (*63*).

Electrical Conductance and Related Phenomena

The equivalent conductance of sodium in ammonia is approximately halved as we proceed from infinite dilution ($\Lambda_0 = 1022$ at $-34°$) to a concentration of 0.02 *N*. At 0.04 *N* the conductance proceeds through a minimum at approximately $\Lambda = 540$, then rises sharply. The experimental curve at low concentrations lies between that predicted by Onsager's equation for a 1-1 and a 1-2 electrolyte. Such a behavior implies interactions leading to the formation of uncharged, nonconducting species and, in view of the magnetic properties, suggests equilibria of the type described by Becker, Lindquist, and Alder (B.L.A.) in their expanded metal model (*1*), as expressed in Equations (4) and (5).

If we assume the applicability of electrolyte theory to metal solutions, then equilibrium constants k_1 and k_2 for equations (4) and (5), respectively, may be evaluated from conductance data. Although it is quite evident that electron conduction proceeds by a tunneling mechanism not involving mass transport in the usual sense (*13*, *58*), we may hope that the conduction mechanism remains essentially unchanged with changing concentration; consequently a mobility decrease with increasing concentration will be experienced by the electron due largely to its interaction with metal ions in accord with Onsager's equation. Support for this proposal comes from the fact that this equation may be used in connection with aqueous solutions of acids, where the proton migrates by a "jump" mechanism. Furthermore the Walden product, $\Lambda_0\eta_0$, for sodium in ammonia at $-34°$, is very close to that obtained with solutions of lithium in methylamine at $-78°$: 2.6 and 2.1, respectively (*2*). This result suggests the conductance to be viscosity dependent, as with ordinary transport.

Evers and Frank (*19*) have used the B.L.A. model in conjunction with a modified form of the Shedlovsky conductance function (*61*) to explain the data of Kraus (*41*) for solutions of sodium in ammonia at $-34°$. Activities calculated by the limiting Debye-Hückel equation were employed for charged species. Later, this analysis was applied with equal success to the conductance of solutions of lithium in methylamine at $-78°$ (*2*). Constants derived from these data are given in Table 1; k_1 refers to equilibrium (*4*), and k_2 to equilibrium (*5*) given above. Constants derived from magnetic data are included; these will be discussed in the section on magnetic susceptibilities.

The constants k_1 in Table 1 are practically the same as those obtained for ion-pairing in solutions of electrolytes in ammonia (*26*) and in methylamine (*20*). These results are encouraging since they imply a fairly normal behavior for dilute solutions of metals. Little may be said regarding k_2, but it is perhaps surprising that k_2 is so low for lithium solutions. The concentrations of the various species computed using the constants in Table 1 for two metal concentrations (*18*) are given in Table 2. It is seen that the predominant species in ammonia is the solvated electron; in methylamine it is the monomer. Whereas in dilute solution in ammonia the dimer is of secondary importance, its concentration increases more rapidly in ammonia than in solutions of lithium in methylamine.

Table 1

System	Method	$k_1 \times 10^3$	k_2	Reference
Constants Derived from Conductance, Transport and Activity Data				
Na in NH_3 ($-34°$)	cond.	7.23	27.0	(*19*)
Na in NH_3 ($-37°$)	trans.	9.2	18.5	(*16*)
Na in NH_3 ($-37°$)	act.	9.6	23.0	(*17*)
Li in CH_3NH_2 ($-78°$)	cond.	0.058	5.42	(*2*)
Constants Derived from Magnetic Data				
K in NH_3 ($-34°$)	B.L.A.	30	98	(*1*)
K in NH_3 ($-34°$)	Deigen	7.9	99	(*11*)

Table 2. Concentration of Species in NH_3 and CH_3NH_2 Derived from Conductance Data N $\times 10^3$

System	(Metal)	$(M^+) = (e^-)$	(M)	(M_2)
Na in NH_3	1.1	1.02	0.07	0.004
	12.4	8.47	1.32	1.28
Li in CH_3NH_2	1.1	0.30	0.72	0.015
	9.8	1.32	6.23	1.14

As noted above, the electron possesses an extra-mobility which is probably to be associated with quantum tunneling from one site to a properly oriented group of molecules in the vicinity of the cavity. As pointed out by Dye *et al.* (*16*), since in principle the Onsager equation does not handle such cases and since it is likely that mechanisms leading finally to metallic conduction might be manifest to some extent in dilute solutions as well, the cation conductance should be a better measure of the free ion concentration than the total conductance. An approximate measure of the cation transference number of sodium was made by Kraus (*42*), employing concentration cells with transference, on the assumption that the activity coefficients in the two-half-cells were unity. These data led correctly to the conclusion that the cation transport number was a decreasing monatonic function of concentration and that the form of the conductance function was determined primarily by the anion conductance. Similar results were found by Klein (*37*) for solutions of lithium in methylamine. Dye *et al.* (*16*), however, have measured the anion transport number at $-37°$ directly in sodium solutions, by the moving boundary method. Their results demonstrate that the anion conductances as derived from Kraus' measurements are too high because activity corrections were neglected; but what is more important, they demonstrated that, using the Shedlovsky equation to describe the conductance

of the free sodium ions, the expanded metal model permitted a satisfactory fit of the data. Constants derived from the data are given in Table 1.

In a second paper (*17*) Dye *et al.* have calculated activity coefficients using transport data from the moving boundary method in combination with emf data from cells with transference (*42*). Using the B.L.A. model allowed extrapolation to infinite dilution and resulted in the values for k_1 and k_2 recorded in Table 1; these values are in good agreement with values derived from conductance and transport data.

Further evidence in support of the expanded metal model is provided by measurements of the effect of temperature on the conductance of lithium in methylamine (*54*). Phenomenologically these results are similar to those observed for solutions of normal electrolytes (*43*). Maxima are observed in the conductance temperature curves for the more dilute solutions. In the case of normal electrolytes, maxima such as these are interpreted as being due to enhanced ion interactions occasioned by a lowering of the dielectric constant. Also, Patterson, Berns, and Lepoutre (*55*) have found from conductance measurements that the addition of salt to solutions of sodium represses the ionization in the expected direction; they conclude that the B.L.A. model is in best accord with the findings.

All the above results are mutually consistent and support the B.L.A. model, which emphasizes the presence of monomers. However, these results do not appear consistent with certain observations regarding the absorption spectrum and do not seem to be entirely in accord with constants derived from magnetic data. A treatment based on e_2^{-2} (am) centers has not been made since this would be exceedingly complex because of additional ion-pairing equilibria.

Magnetic Susceptibilities

The concept of electron coupling (*52*) was first introduced to explain the static field susceptibilities of solutions of sodium and potassium obtained by Huster (*29*) and Freed and Sugarman (*23*). These early results were verified and extended by Hutchison and Pastor using the electron spin resonance method (*31*). The molar susceptibilities increase rapidly with dilution and appear to extrapolate to an infinite dilution value corresponding to an Avogadro number of spins in such a way that, below approximately 0.003 N, the susceptibilities will obey Curie's Law. At higher concentrations the susceptibilities are lower at lower temperatures, but the cross-over point has not been well established (*31*). At −34° the data for sodium and potassium appear to fall on the same curve although in dilute solutions the data for sodium are erratic and scanty, so the curves might diverge. The susceptibilities differ considerably at other temperatures, which points to interactions involving the metal ion. Kaplan and Kittel (*36*) were able to reproduce the data for potassium solutions in a satisfactory manner by using the cavitation model. The best fit was obtained by employing a value of $\Delta H = -2.3$ kcal for the heat of dimerization in equilibrium number (3). This value compares well with the value of $\Delta H = -3$ kcal obtained by Candela (*9*) from measured heats of dilution. However, Patterson (*55*) points out that even a better agreement is obtained if one computes a ΔH based on the B.L.A. model. Values of the equilibrium constant for electron pairing [Equation (3)] recorded by Hutchison and Pastor varied in a regular fashion from about 16 at 0.5 N to about 5 at 0.002 N.

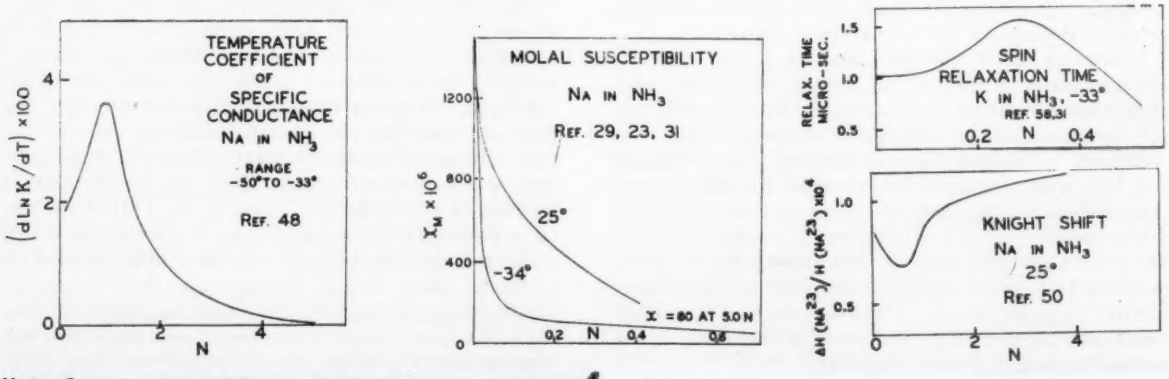
Becker, Lindquist, and Alder, using their expanded metal model (*1*), were able to obtain a satisfactory fit of the data for potassium in the concentration range from 0.005 N to 0.5 N at −34° with constants $k_1 = 0.03$ and $k_2 = 98$ (Table 1). At the ice point these were, respectively, 0.05 and 30; at 25°, 0.02 and 10.

A comparison now seems in order between the results of conductance and magnetic measurements. In making this comparison we shall restrict ourselves to the B.L.A. model. The following factors should be considered:

(1) An analysis of conductivity data was restricted to very dilute solutions; accordingly, the method emphasizes k_1, with k_2 appearing as a correction factor.

(2) Magnetic data up to 0.5 N were used; here k_1 should be relatively unimportant with respect to k_2. Even though the system is undergoing a transition to the metallic state, and the mobility of the electron is increasing rapidly (or the number in a conduction band), the gross effect on magnetic susceptibilities is probably of secondary importance.

(3) Activity corrections for charged species were made in analyzing conductance data; concentrations were employed in deriving constants from magnetic data.



Various Properties of Metal Solutions in the Transition and Metallic Regions

(4) In view of the above considerations, one might infer that values of k_1 from conductance data are to be preferred, while values of k_2 from magnetic data may be more representative.

Unfortunately, because of lack of data, we are forced to compare the magnetic data on potassium with conductance data on sodium at −34°. At this temperature the two metals possess the same magnetic properties, hence we might expect comparable values for the constants. The k_1 value from magnetic measurements is too high to account for the conductance of sodium. The k_2 value from conductance measurements is too low to account for its magnetic properties. However, we do not know the form of the magnetic curves as they extrapolate to infinite dilution from about 0.01 N. Indications are, from optical data, that potassium is more highly associated in dilute solution than is sodium, and probably in the form of dimers. We shall come back to this point when discussing absorption spectra.

A somewhat different approach to a model for dilute solutions has been made by Deigen and Tsvirko (*11*). They call the trapped electron a "polaron" after Landau (*51*) and propose that electrons, associated with M^+ ions, may exist as F_1 centers (corresponding to M) and F_2 centers (corresponding to M_2). A highly elaborate statistical method is used to obtain the distribution of the electrons among the various states. While they do not calculate equilibrium constants, these are implied and may be gotton from their results (*56*). At −33°, they find, to fit magnetic data, that $k_1 = 7.9 \times 10^{-3}$ and $k_2 = 99$; at 0°, the values are 2.2×10^{-3} and 50, respectively. It will be noted that k_1 is in accord with the conductivity data on sodium whereas k_2 agrees with the magnetic results on potassium, referred to above.

Nuclear Magnetic Resonance

McConnell and Holm (*50*) have studied the nuclear magnetic resonance of solutions of sodium in ammonia at room temperature. Unfortunately, because of limitations in the sensitivity of their equipment, concentrations were employed in excess of approximately 0.05 N. Shifts in resonance field strength occasioned by the presence of metal [Knight shifts (*38*)] were substantial in the case of nitrogen. Expressed as the density of the nucleus at the unpaired electron P (^{14}N), this amounts to approximately 13% of that expected for a 2s electron on a nitrogen atom. A considerably smaller density was found in the case of ^{23}Na; but it was sufficient to cause them to conclude that, in the concentration range covered, the electron moves in a highly expanded orbit about the sodium ion. This result is direct evidence of interactions leading to something corresponding to monomers in the B.L.A. model. Above, about 1 N, P (^{23}Na) increases rapidly, suggesting approaching metallic behavior.

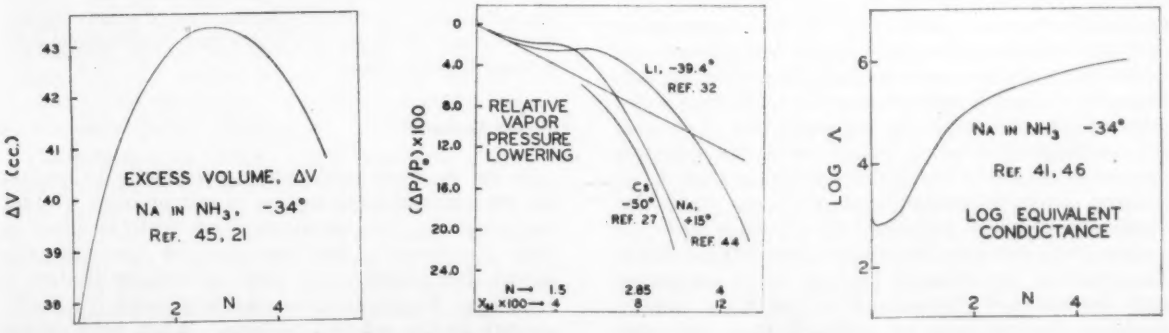
The magnitude and constancy of the ^{14}N hyperfine interaction with changing metal concentration suggest "interaction with unpaired electrons far removed from the sodium ion." The authors suggest that nitrogen atoms may acquire a positive charge by bonding with adjacent molecules through hydrogen and thus act as attracting centers.

Proton Knight shifts were not detected. This fact, coupled with the large values of P (^{14}N), has an important bearing on the structure of these systems. We shall expand upon this feature in the following section.

Paramagnetic Resonance and Electron Spin Relaxation

Reference has already been made to the use of resonance frequency data in calculating equilibrium constants. Measurements of this type are also useful in gaining further detailed information relative to the structure of these systems.

Electrons in cavities or held to some center, as in the species M, should possess a diamagnetism to be associated with their size. This cannot be measured by ac or static field methods alone. However, since in static fields the sum of the para- and diamagnetic contributions is measured, whereas in resonance methods only the paramagnetism is recorded, a comparison of the two methods enables one to estimate the root-mean-square radius. For a concentrated solution (0.5 N), where pairing is virtually complete, Hutchison (*31*) calculated a value of 3.0 Å for the cavity. This value corresponds favorably with estimates of size obtained in other ways for the singly trapped electrons (see below). A striking feature of the esr spectrum is the sharpness of the lines (0.03 gauss). Kaplan and Kittel (*36*) have attributed this to motional narrowing and hyperfine interactions with the solvent nuclei and suggested a cavity model as the most descriptive. They proposed that interactions with protons (directed in toward the cavity) would be more important than interactions involving the nitrogen. This interpretation of line width, as being due to proton interaction, is not in accord with the NMR findings cited above,



Various Properties of Metal Solutions in the Transition and Metallic Regions.

nor is it in accord with more recent studies of electron spin relaxations.

Recent studies by Pollak (*58*), Blume (*6*), and Hutchison and O'Reilly (*30*) have been concerned with an interpretation of line widths and shapes resulting from hyperfine interactions and motional narrowing. They conclude that the relaxation mechanism governing line widths in dilute solutions is the contact hyperfine interaction of electrons with nitrogen nuclei rather than with hydrogen. This premise was supported by measuring relaxation times, using $^{15}NH_3$ in place of $^{14}NH_3$ (*58*) and by demonstrating that substantially no effect was produced when NH_3 was replaced by ND_3 (*30*). According to Pollak, the interaction in dilute and moderately dilute solution is modulated by the relative motion of the electrons and ammonia molecules; an electron tunneling mechanism is proposed as a possible source of this modulation (58). Pollak measured both the spin-lattice (longitudinal) relaxation time, T_1, and the transverse relaxation time, T_2. These he found to be identical in the concentration range from 10^{-4} to 10^{-2} moles of metal per mole of ammonia, and in the temperature range from $-50°$ to $+50°$. On the other hand, Hutchison and O'Reilly found T_1 and T_2 to differ in intermediate ranges of concentration. The reason for the difference between the two sets of data is not immediately apparent.

Absorption Spectra

There have been numerous qualitative investigations of the absorption spectra of alkali metals in ammonia, in amines, and in their mixtures (*24*, *53*, *4*, *22*, *28*). Measurements have been largely limited to concentrations of the order of 1×10^{-3} N or less because of the opaqueness of the solutions. Two broad bands are found; one or both are present depending principally on the nature of the solvent, on the temperature, and to a lesser extent on concentration and on the metal itself. One band occurs in the infra-red region, with reported band maxima ranging from 13,000 to 18,000 Å; the second band is in the visible region, with values of the maximum ranging from 5900 to 7500 Å. For example, dilute solutions of sodium and potassium in ammonia show only the infra-red band (*4*, *14*) at 14,700 Å. Two bands are reported for solutions of lithium in methylamine whereas sodium has only one band in the infrared. In ethylamine and certain diamines the visible band predominates (*22*); in polyethers only the visible band is noted (*15*, *8*, *49*).

Symons and co-workers (*22*, *62*, *63*) have pointed out an interesting correlation between the absorption spectra and magnetic behavior. Where comparative data are available, it has been found that solutions exhibiting absorption bands only in the visible are diamagnetic; if both bands are present or only that in the infrared, the solutions are paramagnetic. Lowering the temperature or increasing the metal concentration appears to favor the visible band at the expense of the infrared band. Considerations such as these led Symons to conclude that the visible band is to be associated with diamagnetic species— probably the dimer, M_2—whereas the infrared band is to be associated with the trapped electron. Since no third band is observed, which may be assigned to a monomer unit, it was concluded that this species could only be present in minor amounts and would not therefore contribute materially to the properties of the systems. However, when sodium iodide was added to a solution of sodium in ammonia, shoulders appeared on the absorption curve at 8000 and 6700 Å; these were attributed respectively to monomers and dimers formed by the repression of equilibria (*4*) and (*5*). It may also be pointed out that Blades and Hodgins (*4*) find a band at 8200 Å for solutions of potassium in methylamine, in addition to one at 6540 Å.

Recently, Douthit and Dye (*14*) have made an exhaustive study of the absorption spectra of dilute solutions of sodium and potassium in ammonia as a function of concentration and temperature. The shape of the absorption curves was identical for the two metals and independent of concentration. A careful search was made for hidden bands that might be associated with monomers or dimers, but none was found.

One significant result of the study was to demonstrate that whereas solutions of sodium conform to Beer's law (concentration range, 3×10^{-4} to 4×10^{-3} N), solutions of potassium show a negative deviation (1×10^{-3} to 1×10^{-2} N). It may be concluded that trapped electrons, which are responsible for the absorption peak at 14,700 Å, are being removed, probably as dimers. Removal as e_2^{-2} (am) species does not seem likely since under these conditions the nature of the positive ion presumably would not be important. This conclusion however does not clarify our position regarding the presence or absence of monomer units, a concept which has been so useful in explaining conductivity data and whose presence is indicated by NMR measurements. On the basis of electrostatics alone, some ion-pairing must occur even in sodium solutions. Reference to Table 2 shows that, according to conductivity data, as much as 20% of the ions are combined into pairs. Accordingly, it is suggested that pairing might best be represented as an ion-pair, $M^+(am)e^-$ (am), with the solvated metal ion remaining outside the cavity. This combination conceivably might absorb much like a trapped electron. Under certain conditions, as at high concentrations or in the presence of an electrolyte having a common ion, the metal ion might slip into the cavity forming monomers M which absorb in the visible ($\sim$8000 Å).

A quantitative study has also been made of the effect of concentration on the absorption spectrum of lithium in methylamine (*60*). A positive deviation from Beer's law was found, using the visible band at 6900 Å. This trend is in the opposite direction from the results discussed above, and may be explained as resulting from the formation of dimers at the expense of other species; this is generally in accord with other observations concerning these systems.

Models Reviewed

So far we have used models principally to explain the phenomenological aspects of our problem without inquiring as to their soundness theoretically or detailing their structures. For example, we are generally agreed that ionization of metal at infinite dilution is complete. The electron therefore must reside in a cavity created in the solvent medium. What then causes stabilization and what are the energetics of this process?

The cavity model was essentially proposed by Ogg (*52*) in which the cavity radius and binding energy was computed using an oversimplified model of an electron in a box. Stabilization was achieved by assuming solvation in the Born sense, but calculations led to entirely too large a radius when compared with the experimental value based on density measurements. This model was then refined by Hill (*25*) and Lipscomb (*49*). More recently a detailed treatment has been made by Jortner (*34, 35*), which is similar to those of Pekar (*57*), Dawydow (*10*), and Deigen (*11, 12*), based on Landau's model (*51*) for possible binding of an electron by polarization of the dielectric medium. Calculations indicate that the electron is confined in a cavity of radius 3.2–3.45 Å, assuming a continuous dielectric medium. This value is well in accord with estimates based on experimental values for the density of these solutions (49). The ground state is the 1*s* state and the infrared absorption band maximum is interpreted as rising from the 1*s* $\rightarrow$2*p* transition of the bound electron, corresponding to an energy of 0.81 ev or a wavelength of $\backsim$15,000 Å. This model, while not detailed, presents the most consistent pattern of size in relation to energetics and correctly predicts the position of the infrared band maximum. In addition, the effect of temperature on the position of the band maximum (*4, 14*) is accounted for by the temperature dependence of dielectric constant and cavity size.

Prior to the measurement of nuclear magnetic resonance, and more detailed studies of electron spin relaxation, it was supposed that the cavity consisted of ammonia molecules with their hydrogen atoms directed toward the trapped electron (*36*). Now it is evident that the nitrogen nuclei interact preferentially with the odd electron, and that the interaction is relatively independent of metal concentration (*50, 58, 30*). Blumberg and Das (*5*) have used the B.L.A. model to interpret the NMR data of McConnell and Holm (*50*), and have concluded that the experimental results may be explained satisfactorily without recourse to the trapped electron concept. It must be remembered, however, that these data were obtained in relatively concentrated solutions where association might be expected to be substantial. The theory however does predict the results to be expected as we approach infinite dilution; further data in dilute solutions would be most welcome.

The existence of monomers in the B.L.A. sense seems questionable, at least for dilute solutions in ammonia in view of the optical studies noted above (*14, 33*). A view more consistent with conductance and optical studies would require something approaching ion-pairing. It is conceivable however that at higher concentrations and in solvents of lower dielectric constants, like the amines, where structural factors also must become important, we might possibly obtain monomers in the B.L.A. sense.

The existence of dimers seems reasonable. But it must be pointed out in this connection that the high value of the absorption energy for the dimer (15,000 cm^{-1}) in solution, compares favorably with the energy for the $\epsilon_u^+ \leftarrow \epsilon_g^+$ transition observed for gas molecules (*14*): 14,659 cm^{-1} for Na_2 and 11,620 cm^{-1} for K_2. This suggests that the dimer in solution is essentially the same as the gas phase dimer and may not be the loosely bonded, expanded model suggested by Becker, Lindquist, and Alder.

The Transition Range of Concentrations

The properties of dilute metal solutions apparently may be described in terms of ions, trapped electrons, ion-pairs or "atoms," and "molecules." Such solutions may be described as "quasi-electrolytic." In contrast, concentrated solutions are definitely metallic.

The approach to the metallic state becomes most evident through conductance studies and particularly through measurements of transport numbers. The latter affords ample evidence that the transport mechanism, leading to metallic-like conduction, extends well into the dilute range, below approximately 0.02 *N*. While bulk magnetic properties are not so sensitive to this transition, nuclear magnetic resonance and electron spin relaxations indicate abrupt changes in the neighborhood of 0.5 *N*; these may be associated with the onset of the metallic state. At this concentration it may be computed that metal atoms on the average are approximately 10Å apart; electron wave functions can therefore overlap producing conduction bands. Accordingly, we shall define the transition range as lying between 0.04 *N* and approximately 1.0 *N*. Although this selection is somewhat arbitrary, it will be found that in this range many of the properties vary with concentration in such a way that trends in their variations are reversed. Inflection points are obtained in plots of these properties versus concentration, and maxima or minima in their variations, as vapor pressure (*44*). Two liquid phases are found with certain metals at lower temperatures (*48*).

In so far as possible, the various properties in the transition and metallic regions are compared in Figure 1 where the concentration units are expressed as mole fraction of metal, X_m, or normality, *N*; most of the data are for solutions of sodium in ammonia at $-34°$. We shall now consider the behavior of various properties.

Conductance (*46, 47*). After proceeding through a minimum at 0.04 *N*, the equivalent conductance commences to rise almost exponentially. What is more important, the relative change in specific conductance per relative change in concentration, $d \ln k / d \ln c$, increases markedly in the intermediate concentration range, which means that the conductivity is increasing at an increasing rate. The function then proceeds through a maximum at about 1 *N*, and decreases to a constant value at approximately 2.5 *N*; although the conductivity in the meantime is increasing, it is doing so at a decreasing rate.

Temperature Coefficient of Conductivity (*48*). The temperature coefficient of conductivity, dk/kdt, where *k* is the specific conductance, represents the relative change of conductivity per degree. In the dilute region for sodium, it is about 2% per degree. Thereafter it increases to a maximum of 3.6% at a concentration of approximately 0.7 *N* (*48*). A behavior such as this, taken in conjunction with the conductance itself, points to a drastic change in the conductance mechanism. In the more dilute region we may postulate that electron conduction proceeds by a tunneling mechanism (*13*). As the concentration is increased, some electrons may be transferred to a conduction band (*33*); this would lead to an increase in the average

temperature coefficient and would account for the observed increase in conductivity itself. After the maximum, the temperature coefficient starts decreasing; this provides evidence for the beginning of true metallic conduction; some electrons may now conduct without excitation to the conduction band.

Vapor Pressure. Cesium excepted (*27*), deviations from Raoult's law are positive in dilute solution. With both lithium (*32*) and sodium (*44*) the rate of decrease of vapor pressure with increasing metal content drops sharply, then reaches a minimum. With sodium at 15° this occurs at approximately 1.5 *N*. The pressures now are much too high for ideal solutions. Apparently solvent is being excluded due to the formation of a metal-like lattice. At lower temperatures liquid phase separations are noted (*48*); in the case of sodium the upper consolute temperature occurs at −41.6° and a metal concentration of 1.5 *N*. A major change in structure is indicated.

Knight Shifts. The NMR shift (*50*), while small for sodium, depends on the metal concentration. It passes through a minimum at about 0.75 *N* then increases quite rapidly. The initial decrease might be attributed to aggregation in the sense of pairs; after the minimum, electrons are freed and shared by sodium ions of the liquid lattice. The change in electron spin resonance line widths agrees with these views.

Thermoelectric Power. The termoelectric power decreases continually with increasing concentration (*13*). However, a plot of the data shows a slight variation at intermediate concentrations, with a small change in slope at approximately 0.75 *N*.

Volume Excess (*45, 21*). An expansion is occasioned by the solution of metal. Experimentally this volume change, ΔV, is defined as:

$$\Delta V = \frac{\text{Volume of solution} - \text{Volume of components}}{\text{Number of moles of metal}}$$

This function appears to pass through a minimum in the region of the conductance minimum (*21*). Thereafter it rises to a maximum in the metallic region at approximately 3.0 *N*. It might be assumed that the increase in ΔV tells us that we are breaking down the structure of ammonia; evidently this has something to do with the formation of a metallic lattice. In the same concentration range the solvent is being excluded, as evidenced by the excess vapor pressures.

The Metallic Region

We shall state arbitrarily that the metallic region extends from above 1.0 *N* to saturation. Here the vapor pressure, after passing through a flat region at approximately 1.0 *N*, decreases sharply. Undoubtedly a good proportion of the solvent is being bound to the metal ions with small tendency to escape. Apparently, interaction of solvent molecules with metal ions grossly outweighs their exclusion by the metallic lattice, an effect which predominates at lower concentrations. At saturation, for example, there are roughly 5.5 molecules of solvent per atom of metal, and the vapor pressure is extremely low.

The excess volume, which increases up to approximately 3 *N*, commences to decrease. This decrease could now result from close-packing about the metal ions due to solvation effects.

The temperature coefficient of conductance decreases rapidly, reaching the very small value of 0.005% at saturation. Apparently this would become negative if concentrations could be increased slightly. The thermoelectric power seems to behave as if we had a degenerate Fermi-Dirac electron gas (*55*). Meanwhile the conductance has increased to the order of 1×10^6 Kohlrausch Units, and the magnetic susceptibility approaches values corresponding to the pure metals. We have now reached the metallic state where all properties vary with concentration as those of a metal in a dielectric medium.

Summary

Particular emphasis has been placed on the behavior of dilute solutions, in this review of the properties of solutions of metals in amine solvents. It is generally accepted that at high dilution, in a solvent such as ammonia, ionization of the metal is complete, yielding metal ions and electrons. The latter are "solvated" by or "trapped" in the solvent medium. Two models have been proposed to interpret the fate of the electron as the metal concentration is increased. One model proposes that trapped electrons, e^- (am), pair up with spins opposed yielding a diamagnetic species consisting of two electrons in a cavity, e_2^{-2} (am). The second model differs from the first in that the metal ion is involved in equilibrium reactions with the trapped electron and thus contributes materially to the state of the system. Equilibria are postulated involving the solvated electron e^- (am), and solvated metal ion, M^+ (am), to yield a monomer, M, and a diamagnetic dimer, M_2. The relative merits of these models are discussed as they may be used to interpret magnetic, electrical, and optical data. At high solute concentrations, metal solutions exhibit true metallic behavior. The transition region between the quasi-electrolytic and metallic states is discussed qualitatively in relation to the phenomenological aspect.

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The Franklin Institute Chemistry Exhibit

A design for learning

At its inception the Franklin Institute Science Museum represented a departure in the educational services of the Institute, services which have continued since 1824. For the first time in this country, visitors to a technical museum could learn by doing. Scores of visitor-operated exhibits were available. Prominent among the exhibit halls was the Hall of Chemistry where, over the years, 22 different exhibits were set up to demonstrate fundamental chemical phenomena. Among the particularly effective exhibits were the combustion of iron in pure oxygen, the changing of oxygen into ozone, the electrolysis of tin chloride, and the electroplating of copper. The focal point of the Hall of Chemistry was a periodic table of the elements, containing samples of many elements.

By 1952, the rapid changes in chemical knowledge and the lack of funds to keep the exhibits up to date and in good working order brought about the demise of the Hall of Chemistry. The loss was immediately felt by visitors. Their questions to museum personnel and their letters to museum officials indicated an active interest in chemistry which could best be satisfied by another exhibit. In 1954, under the aegis of A. C. Carlton, late Director of the Science Museum, plans were formulated for a new exhibit. With the aid of an *ad hoc* Advisory Committee to the Franklin Institute, established (on invitation) by the Philadelphia ACS Section, working plans were completed. These consisted of broadly outlined topics which the Advisory Committee felt could be included in a comprehensive exhibit

with the objectives of:

Stimulating young people to take up careers in chemistry;
Providing for students educational material in the basic principles (and some applications) of chemistry;
Providing information for the casual adult visitor about fundamentals of chemistry.

By the summer of 1957, under the tireless, dedicated leadership of Dr. J. Bennett Hill of the Sun Oil Company and Dr. Charles S. Venable of the American Viscose Corporation, retired Directors of Research in their respective corporations, a fund-raising campaign began. Two years later sufficient funds had been contributed by the chemical industry and friends of the Franklin Institute to allow the beginning of the final planning and construction of the exhibit. In late August, 1959, the authors were hired as consultants to the Institute for the purpose of creating the exhibit. Each of us is a teacher of chemistry; the fact that perhaps half a million people would visit the exhibit yearly was therefore not just a challenge to us, but, rather, a mandate to create an exhibit which would be useful and educational within the framework of its three objectives, and in addition would be dramatic and informative.

At the outset it was decided that the exhibit should be designed for learning and so arranged that logical sequence of facts and displays should be of prime importance. To this end, exhibits to demonstrate the following sequence of topics were developed: The Structure of Matter, The States of Matter, The Interactions of Matter, Inorganic Chemistry, Organic Chemistry, Biochemistry, and Radiochemistry.

The Structure of Matter. The visitor is introduced to the exhibit and to chemistry by listening to a short tape-recorded discussion of what chemistry is all about. Integrated with the tape is a series of animated slides which are projected on a screen at appropriate places to reinforce visually what is being explained orally. In this introductory panel the visitor is given a simplified picture of the nuclear atom which although, as he is told, "is by no means the totally correct one, still has much merit. Such a picture of atomic structure parallels that of our solar system." Emphasis is then given to the periodic nature of electronic configuration and the chemical implications of this. This first panel ends with the visitor reading Benjamin Franklin's comment to Joseph Priestley which aptly fits the contemporary chemical scene:

The rapid progress true science now makes, occasions my regretting sometimes that I was born too soon. It is impossible to imagine the heights to which may be carried, in a thousand years, the power of man over matter.

The visitor is next introduced to chemical bonding and some of its consequences. Again the description is an oral one, heard through a telephone activated by a push button. The visual reinforcement in this section consists of back-lighted panels, and operating or static exhibits lighted or activated in sequence with the taped discussion. A short discussion of ionic bonding is first given with the formation of sodium chloride from the elements as an example. Care has been taken to give the correct impression that the ion sizes differ from atom sizes. A model of the sodium chloride lattice is presented and discussed, followed by a demonstration of the electrical conductance of a molten salt as opposed to the non-conductance of a dry salt. Then follows a discussion of covalent bonding using the formation of water from the elements as an illustration. A demonstration of the electrical conductance of salt water as opposed to the non-conductance of "pure" water[1] leads directly to a discussion of polarity and polar and non-polar covalent molecules. A diagrammatic explanation of polar molecules uses hydrogen chloride as an example, while a model of carbon tetrachloride is used to illustrate non-polar molecules. The panel on chemical bonding ends with a static exhibit of the effect of polarity of molecules on solubility with potassium dichromate and iodine as solutes and water and carbon tetrachloride as solvents.

Figure 1 shows the ionic bond unit and part of the covalent bond unit of the chemical bonding section. (It might be observed here that many visitors neglect to look at the panels while listening to the taped discussion.)

The States of Matter. With the same technique of taped discussion and synchronized exhibits the visitors are first introduced to crystal symmetry (models of symmetry classes and natural examples are shown). For dramatic effect he next sees a rotating "chemical garden" of simple silicates. There follow three exhibits on some properties of liquids, utilizing surface tension (mercury versus colored water), fluorescence (dramatically illustrated with fluorescein) and index of refraction (using pyrex "disappearing" in trichloroethylene).

The gaseous state is next briefly discussed in terms of the familiar porous cup diffusion experiment using helium gas. A final panel on the changes of state which matter may undergo completes the topic of the structure of matter. In this last panel the visitor sees an iodine sublimation exhibit and the crystallization of sodium thiosulfate pentahydrate from a supersaturated solution.

The Interactions of Matter. This section is introduced by a short taped discussion of the difference between physical and chemical change. Here again, a synchronized set of slides provides visual emphasis. In this brief introduction the concepts of energy of chemical reactions and of chemical equations are presented.

The visitor is then on his own for a while as he may view five exhibits of different kinds of chemical reactions each explained in writing. The first is a static mock-up of Priestley's discovery of oxygen. The next four are visitor-operated exhibits. The first of these, and probably the best-liked part of the entire room, is an exhibit

The cover

When a family from Yonkers, N. Y., makes a pilgrimage to the city of the Liberty Bell, they enjoy learning modern science as well as American history. The console in front of the children bears buttons identified with questions. Pushing a button answers the question by selectively lighting positions on the table.

Readers will note the only moderate optimism of the designers, who left space for elements 104–?, but did not have the panel space cut out.

[1] Apparatus designed by SCHMUCKLER, J., see J. CHEM. EDUC., **33**, 506 (1956).

redeemed from the former Hall of Chemistry. This is an exhibit in which water is electrolyzed and the hydrogen and oxygen formed is noisily exploded. Next follows the displacement of copper by iron, galvanic action (Daniell Cell) and the photochemical reduction of thionine by iron(II).

There follows a taped discussion and slide sequence on catalysis ("chemical bulldozers") and a static, though highly dramatic, exhibit on the catalytic oxidation of methanol.

Figure 1. Unit on chemical bonding.

Inorganic Chemistry. The section on inorganic chemistry is introduced by an *apologia*, summarized in its last sentences:

> The modern inorganic chemist is not merely interested in the tabulation of descriptive data. He seeks to explain, on the basis of sound chemical theory, why such data exist. . . . It should be emphasized that though the unit on inorganic chemistry you are about to visit is essentially designed from the classical (descriptive) point of view, most of the modern developments in the field are based on theoretically sound experiments, rather than on accidentally discovered facts.

The unit on inorganic chemistry is divided into descriptive sections on metals and alloys, including a discussion of the properties of metals based on the simplified picture of metal crystals consisting of "closely packed nuclei in a sea of outer electrons"; on non-metals, including a discussion of allotropy (carbon and phosphorus as examples); and on inorganic compounds with particular emphasis on applications in the fields of health, agriculture, and construction. Throughout the entire section a taped discussion is illustrated with keyed-in diagrams, samples, models, etc.

Organic Chemistry. The first unit of this section is a taped discussion with synchronized slides of a very brief introduction to aliphatic organic chemistry. The visitor is exposed to the principles of carbon-carbon bonding and functional groups primarily. The next unit of the section is devoted to a consideration of giant molecules. A brief discussion of the orientation of molecular chains as being vital to helping establish the final useful properties of polymeric materials leads into a discussion of natural rubber and the vulcanization process. A "rubber engine"[2] activated by a heat lamp ends this unit.

The next unit describes giant inorganic molecules, the mineral silicates, and leads into a discussion of the silicones. The unit ends with a continuous demonstration of the synthesis of nylon.[3] Lest the reader become confused at the inclusion of nylon with a discussion of silicones, it should be stated that this demonstration serves to conclude the three units on giant molecules. ("You have heard and seen much concerning giant molecules. Now you will see one giant molecule being made.")

The next unit describes ion-exchange resins and the problem of hard water, and ends with a demonstration of the softening of water by such resins. (The softening is observable by comparing meter readings of ppm-ion content before and after the water passes through a resin column.) This unit is logically followed by one in which soaps and synthetic detergents are considered, emphasizing the mechanism of detergent action in terms of the structure of detergent ions. A simple demonstration of the effectiveness of a syndet in hard water is included.

The next unit is entitled "Techniques of the Organic Chemist." Here the visitor sees spinach being extracted in a Soxhlet Extractor; the intensely green extract is then concentrated by distillation of the extractant through a Snyder Column (effective from both the visual and aural sense); the results of passing the concentrated extract through a column filled with alumina ends the sequence. Throughout this unit the visitor reads descriptions of the various separation techniques.

The final unit in the organic chemistry section is entitled "The Creativity of Organic Chemistry." This is a static exhibit depicting the use of benzene in the production of such basically different materials as nylon, indigo, DDT, aspirin, polystyrene, Bakelite, sulfanilamide, and sodium dodecylbenzenesulfonate.

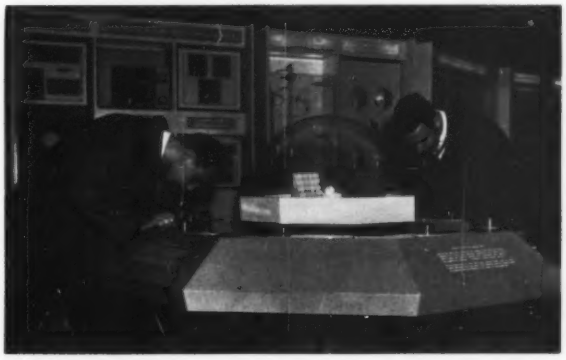

Figure 2. The odor bar.

The favorite unit in the organic chemistry section is actually an adjunct to it and not part of the sequential discussion. This is the appropriately-named "Odor Bar." At this octagonally-shaped exhibit the visitor gets to smell what should be a familiar odor (acetic acid,

[2] The rubber engine is based primarily on the Wiegand Rubber Heat Engine (Entropy Wheel), and specifically on the design of the model shown at the Brussels World Fair and created by the E. I. du Pont de Nemours Co., Inc.

[3] MORGAN, PAUL W., AND KWOLEK, STEPHANIE L., J. CHEM. EDUC., **36**, 182–4 (1959).

vanillin, paradichlorobenzene, cinnamaldehyde, methyl salicylate, isopropyl alcohol, isoamylacetate, and camphor), guess at what it is and then determine how correct he was. This exhibit (called "Chemical Scents and Chemical Sense") is so engrossing that, unfortunately, few visitors to it notice the attached exhibit on selective dyeing. In the latter can be seen a mock version of the selective dyeing of a material which is a composite of four fibers in a single dye bath. An attractive plaid design is a result. In Figure 2 may be seen the "Odor Bar" and the demonstration of selective dyeing and, in the background, some of the units of the sequential discussion of organic chemistry.

Biochemistry. The section on biochemistry is devoted to a relatively high-level discussion of proteins, enzymes, metabolism, ATP, chemotherapy, and DNA. The taped discussion and keyed-in back-lighted panels introduce the visitor to such items as the peptide linkage, the sequence of amino acids in insulin, enzyme catalysis, metabolic maps, polynucleotides, and the hydrogen bond. A dramatic adjunct to the section is a 4-ft-high model of a section of the DNA double

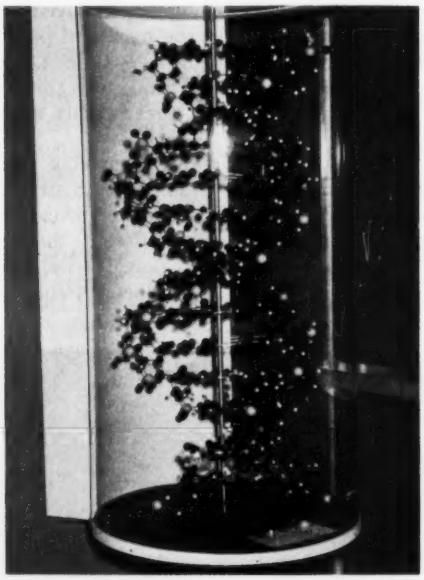

Figure 3. A model of deoxyribonucleic acid.

helix[4] (see Fig. 3). The section ends with three demonstrations. The first is entitled photosynthesis; the oxygen evolved by an aquatic plant exposed to light is visibly evident and is contrasted with the lack of such evolution when the same plant is kept in relative darkness. The second is another living experiment in which the growth of gibberellin-treated and non-treated bean plants is observed and recorded. The final demonstration is entitled "Techniques of the Biochemist." Currently on display is an operating zone-melting apparatus on loan from the Fisher Scientific Company. It is hoped that future apparatus in this unit will be less subtly associated with biochemical researches (perhaps a Warburg apparatus or electrophoresis equipment).

Radiochemistry. It may be recalled that throughout most of the sequential topics discussed so far, the primary technique employed is a taped discussion with synchronized visual effects. In this last section, the discussion was treated solely by printed material and diagrams. The first unit is an explanation of the nature of radioactivity. With it is associated a visitor-operated demonstration of the effects of a magnetic field on beta particles, followed by units on "Radiation and Matter" (including an operating thermos bottle cloud chamber and a visitor-operated shielding experiment), "Isotopes and Their Uses" (including a mock demonstration of the use of radioisotopes to locate liquid levels), and finally "Nuclear Energy: Fission" (no demonstrations).

Two final aspects of the Chemistry Room should be covered. As in the former Hall of Chemistry, the central feature of the room is the periodic table of the elements. The current version (see Fig. 4) is electronically controlled by a small console before it, on which nine questions appear relating to the elements. When the visitor operates the console, the answers to the questions flash on the chart. A sample question is, "Which elements are gases at room temperature?" A final important feature of the room is an alcove which has been equipped with a demonstration table and blackboard. From time to time qualified lecturers augment the material to be learned in the room with lecture-demonstrations here.

Since many of the visits to the Franklin Institute Science Museum are made by classes of students, provision has been made for switching taped discussions, normally heard individually through telephones, to a loud-speaker system.

The new Chemistry Room has met with a good measure of success; Museum officials plan to pattern future exhibits on it.

Acknowledgment. We would like to acknowledge the invaluable aid and encouragement of Robert W. Neathery, Head, Franklin Institute Science Museum. Staff members of the W. E. Sparks Studio, Inc., patiently learned a great deal of chemistry and helped, through their creative design, to make the Chemistry Room truly a design for learning.

[4] This model was created for the Chemistry Room by Mr. A. A. Barker, 41 Stretten Avenue, Cambridge, England.

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A Model for Optical Rotation

THE rotation of the plane of polarization of light passing through a medium is called optical rotation. Substances which have this property are said to be optically active. Optical rotation (*1*) was discovered by Biot in 1812. In 1825, Fresnell (*2*) correctly described optical rotation as a difference in refractive index for left and right circularly polarized light. The Fresnell equation is

$$\phi = \frac{\pi}{\lambda}(n_l - n_r) \tag{1}$$

where λ is the wavelength, in vacuum, of the incident light, n_l and n_r are the refractive indices for left and right circularly polarized light respectively, and ϕ is the rotation in radians per unit length in the units of λ, of the active medium. There is also a corresponding difference in the absorption of left and right circularly polarized light in optically active media. This was discovered in crystals by Haidenger (*3*) in 1847 and in liquids by Cotton (*4*) in 1895. This phenomenon is called the Cotton effect and may be expressed as,

$$\theta = \frac{\pi}{\lambda}(K_l - K_r) \tag{2}$$

θ is called the ellipticity per unit length. K_l and K_r are the imaginary parts of the complex refractive indices which are a measure of the absorption by the relation

$$I_l = I_{l_0}\, e^{-4\pi K_l Z/\lambda}$$

I_l is the intensity of the left circularly polarized light after it has traversed a distance Z of the optically active media when I_{l_0} is the initial intensity.

Optical rotation in crystals may depend on the arrangement of the atoms or molecules along spiral paths in the crystal latice, as in the case of quartz. Optical rotation arises when plane polarized light is propagated through a medium along the direction of an applied magnetic field. This is the Faraday effect, which all substances show, and arises from the splitting of spectral lines in a magnetic field, in analogy with the Zeman effect. In isotropic liquids which are optically active, the activity must arise from the asymmetry of the individual molecules. It is this natural optical activity which we wish to consider.

The first molecular model of optical activity was proposed in 1892 by Drude (*5*). In his model, an electron, or "charged particle," was constrained to move along a helical path while oscillating around an equilibrium position. The expression Drude derived may be written

$$\phi = \sum_i \frac{R_i}{\lambda^2 - \lambda_i^2} \tag{3}$$

where λ is the wavelength of the incident light, λ_i is the wavelength corresponding to a particular vibration of the electron along the helix, and R_i is a parameter whose numerical value and sign is a characteristic of the periodic motion, i. The sum is taken over all periodic motions of the molecule. The form of the Drude equation and the concept of periodic motion are essentially correct; the wave-length dependence of optical rotation data is fitted well by the equation when measurements are made at wavelengths sufficiently distant from the absorption wavelengths, λ_i. Although the Drude equation is useful, the particular model he used was shown by Kuhn in 1933 (*6*) to exhibit no optical activity when treated rigorously. Kuhn (*7*) proposed that two interacting oscillators were necessary to produce optical activity. These oscillators must move in different directions and must oscillate around different centers. This model of coupled oscillators gives the same form of equation for optical rotation as does Drude's.

The quantum mechanical expression for optical rotation was derived in 1928 by Rosenfeld (*8*). This formulation was subsequently used in calculations of optical rotation by Boys (*9*) in 1934, Kirkwood (*10*) in 1937, and by Condon, Altar, and Eyring (*11*) in 1937. These calculations were based on different models; Kirkwood's were based on a coupled oscillator model and those of Condon, Altar, and Eyring on a single oscillator extension of Drude's model. These two sets of calculations agreed, however, that the absolute configuration of secondary butyl alcohol was that assigned by the Fisher convention, after a correction of a sign error in Kirkwood's calculation (*12*). These models were discussed and a derivation of the Rosenfeld equation was given in a review article by Condon (*13*) in 1937.

A thorough account of the experimental and empirical side of the subject was given by Lowry (*14*) in 1935. The interpretation of the experimental facts concerning optical activity with relation to temperature, internal rotation, and solvent effects was given by Kauzmann, Walter, and Eyring (*15*) in 1940. The current uses of optical activity have been given by Djerassi (*16*).

The work on optical rotation in 1937 was concerned with the determination of the absolute configuration of molecules; either to prove or to disprove the Fisher convention. Although these calculations showed that the Fisher convention was correct, they were not completely unambiguous. The Fisher convention has recently been shown to be correct by the X-ray determination of the crystal structure of sodium rubidium tartrate dihydrate. By using X-rays with a frequency on the absorption edge of the rubidium atom, a phase lag is introduced into the dispersion and it is

EDUCATION

possible to tell the distance between atoms and also the sequence in which the atoms occur, which is all that is necessary for the determination of the absolute configuration. This work was done by Bijvoet (*17*). A similar treatment was carried out by Saito (*18*) on the absolute configuration of the ethylenediamine cobalt complex ion. The current interest in optical rotation is to determine the molecular mechanisms which produce optical rotation and to devise a method of determining the absolute configuration of organic molecules without going through the laborious process of relating them to other molecules with known configuration. The knowledge of absolute configurations is a great help in the study of optical activity, since the calculated rotations can be checked with known molecules and the important terms recognized.

Measurements and Interpretation

Optical rotation measurements, when taken as a function of wavelength, show maximum and minimum values, referred to as anomalous rotatory dispersion. The wavelength dependence can be written as a sum of terms, each one corresponding to an electronic transition in the molecule. For example, the curve shown solid in Figure 1 can be decomposed into the sum of the dotted curves, each of the dotted curves corresponding to a term in the Drude equation. It is then convenient to consider each of these parts individually. A part of the rotation arising from a single electronic transition is shown in Figure 2. The solid line is the measured curve and the dotted portion is the curve predicted by a single term in the Drude equation. The differences between these curves can best be thought of as arising from the classical radiation damping, which is appreciable only near the absorption wavelength. The radiation damping can best be expressed by using the imaginary complex rotation, in which case the rotation is the real part and the absorption, the Cotton effect curve, is the imaginary part of the complex expression. In Figure 2, λ_0 is the wavelength of the electronic transition and ϕ_1 and ϕ_2 are the extremum values of the rotation at the long and short wavelength sides of the absorption band respectively. The absorption band at λ_0 is a characteristic

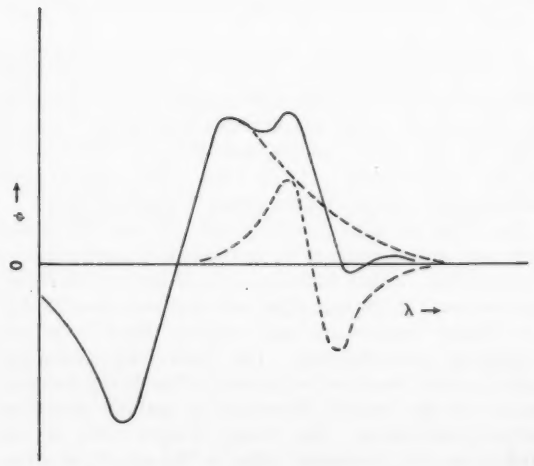

Figure 1.

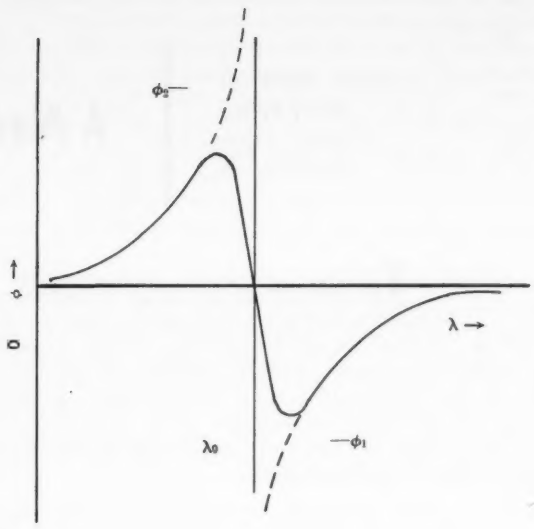

Figure 2.

of a particular electronic transition in the molecule. This transition can be considered localized in a particular group of the molecule. This group is called the chromophoric group. For example, the absorption and dispersion band which occurs at about 2900 Å in optically active, saturated ketones can be ascribed to a definite transition in the carbonyl group. The magnitude of the rotatory power for a transition is conveniently measured by the difference in rotation between the long and short wavelength extremum values, $\phi_1 - \phi_2$. This magnitude will depend on the environment of the chromophoric group. If the groups in the molecule which are near the chromophoric group, called the vicinal groups, are located so as to give a plane or center of symmetry around the chromophoric group, the rotation will be zero. Because of internal rotation about bonds, the chromophoric group will usually be in an asymmetric environment or field; however, the time average of this field will be symmetric and the rotation will vanish unless some group in the molecule prohibits this internal racemization. The role of the asymmetric center in organic molecules is to remove this internal racemization and thus create a time average asymmetric field at the chromophore. The chromophoric group may be several atomic distances from the asymmetric center. Except in the hydrocarbons, the effect of the chromophoric character of the asymmetric carbon is usually obscured since its absorption lies in the far ultraviolet. Dispersion measurements are conveniently made for transitions in the visible or near ultraviolet only, above about 2500 Å.

The Rosenfeld Equation

The expression for optical rotation is due to Rosenfeld. A convenient form of this equation is given by Eyring, Walter, and Kimball (*19*), where ϕ is the rotation in radians per centimeter and the transitions are from the ground state of the molecule, designated by the eigenfunction Ψ_0, to the excited states, designated by the functions Ψ_i.

$$\phi = \frac{16\,\pi^2 N}{3\,ch}\left(\frac{n^2+2}{3}\right)\sum_i \frac{R_{i0}\nu_{i0}}{\nu_{i0}^2 - \nu^2} \qquad (4)$$

Here R_{i0} is the imaginary part of the scalar product of the matrix elements for the electric and magnetic moments,

$$R_{i0} = Im\ \{(\Psi_0|R|\Psi_i)\cdot(\Psi_i|M|\Psi_0)\} \qquad (5)$$

N is the number of active molecules per cubic centimeter, n the index of refraction of the solution, ν_{i0} the frequency of the transition from the state Ψ_0 to the state Ψ_i, and the sum is taken over all excited states, i. Using this equation and the complete set of eigenfunctions for the molecule, one can calculate the optical rotation. Unfortunately, the complete set of eigenfunctions is not known for molecules in general so that a straightforward calculation is impossible. It is convenient, for purposes of calculation, to use a model which describes the features of the molecule which are essential for optical rotation. From the Rosenfeld equation we can see that the condition for optical rotation is that the imaginary part of the scalar product of the magnetic and electric dipole moments be different from zero for the transition $\Psi_0 \rightarrow \Psi_i$. The scalar product of these two matrix elements cannot be different from zero, when the chromophoric group has a plane or center of symmetry. We can see this by considering a chromophore which is symmetric with respect to the plane defined by the Y and Z coordinate axes. The eigenfunctions may be classified as odd or even with respect to reflection in this plane, i. e., the eigenfunction either changes sign or does not at the plane. The X component of R, the electric dipole moment operator, is odd with respect to reflection in this plane and will only have a non-vanishing matrix element between odd and even states. The X component of the magnetic dipole moment operator, M, is even with respect to this reflection and will only have matrix elements between two even or two odd states. An analogous discussion holds for the Y and Z components. Thus, there are no two states for which the scalar product of the electric and magnetic dipole matrix elements does not vanish when the chromophore possesses a plane of symmetry.

The transitions which give rise to the optical activity will occur in all molecules in which the particular chromophoric group exists. The transition will show rotation only when the chromophore is in an asymmetric environment. The frequency of transition will change only slightly due to the neighboring groups, so that the transition may be classified as to frequency and the transition involved, with respect to the local symmetry. Since most chromophoric groups will have local symmetry, the transition will be primarily either electric-dipole-allowed, between an odd and an even state, or magnetic-dipole-allowed, between two odd or two even states. The optical rotation will arise from the perturbation of these symmetry states by the vicinal groups to add in the conjugate transition matrix. An optically active transition will then be either electric-dipole-allowed and the magnetic moment to states brought in by the perturbation, or magnetic-dipole-allowed and the electric dipole to states brought in by perturbations. These two cases may be distinguished by the absorption spectra at the frequency of the transition. If the absorption is small, the transition will be magnetic-dipole-allowed and if the absorption is large, the transition will be electric-dipole-allowed.

The Harmonic Oscillator Model

The simplest model which exhibits all features of optical activity was shown by Condon, Altar, and Eyring (*11*) to be an anisotropic, asymmetric, harmonic oscillator. We will show how this model may be used to predict absolute configuration of optically active molecules when the rotatory dispersion data are known.

The potential used by Condon, Altar, and Eyring was

$$V = {}^1/_2\,K_1x^2 + {}^1/_2\,K_2y^2 + {}^1/_2\,K_3z^2 + Axyz \qquad (6)$$

where x, y, and z are three orthogonal coordinates. The condition for anisotropy is that the restoring force in each of the three directions be different, or $K_1 \neq K_2 \neq K_3$. The term in $Axyz$ provides the necessary asymmetry. The initial eigenfunctions are calculated from the harmonic oscillator potential. These initial eigenfunctions will give the energies and transitions of the system. The term in $Axyz$ is treated as a perturbation. The terms added to the initial eigenfunctions by the perturbation are just sufficient to produce optical rotation. The expression obtained by Condon, Altar, and Eyring for the optical rotation when the oscillator is in an initial state characterized by the harmonic oscillator quantum numbers (n_1, n_2, n_3), for excitation in the X, Y, and Z directions respectively, is

$$\phi = \frac{Nhc^2A}{16\,(2\pi)^3\,\mu^3\lambda^2}\left(\frac{n^2+2}{3}\right)\cdot\left\{\left(\frac{1}{\nu_2}-\frac{1}{\nu_3}\right)\frac{n_1+n_2+1}{(\nu_1+\nu_2)^2-\nu_3^2}\right.$$
$$\left[\frac{1}{\nu_3^2-\nu^2}-\frac{1}{(\nu_1+\nu_2)^2-\nu^2}\right]+\left(\frac{1}{\nu_2}+\frac{1}{\nu_1}\right)\frac{n_2-n_1}{(\nu_1-\nu_2)^2-\nu_3^2}$$
$$\left.\left[\frac{1}{\nu_3^2-\nu^2}-\frac{1}{(\nu_1-\nu_2)^2-\nu^2}\right]+\text{terms in the cyclic permutation of the indices, 1, 2, and 3.}\right\} \qquad (7)$$

In this equation μ represents the reduced mass of the oscillator; $\nu_i = ({}^1/_2\,\pi)(K_i/\mu)^{1/2}$ are the frequencies corresponding to the transitions in each of the principal directions. It is interesting to note that, using the correspondence principle in the limit of large quantum numbers, Planck's constant disappears. Thus the model shows optical rotation in the classical limit.

From equation (7) we see that the rotation is proportional to the parameter A, the differences between the frequencies of the transitions in the various directions, and the quantum numbers of the initial state. The rotation vanishes if any two of the transition frequencies are the same. The model is somewhat artificial, there being no molecules or chromophoric electrons obeying such a potential; however, the rotation for actual chromophoric electrons will be well expressed by equation (7) with an appropriate interpretation of A. The exact form will be similar with respect to the transition frequencies, the energies, the initial quantum numbers, and the sign of the parameter A. We can determine the sign of rotation by fitting this fictitious one-electron potential to the actual potential and using equation (7).

To fit the potential of equation (6) to the molecule

we chose the center of the coordinates in the chromophoric group. To determine the harmonic oscillator potential in each of the three principal directions, we consider the effect on vicinal group electrons of a transition in the chromophoric group. These will usually be the electrons of the closest neighbors and will be affected by the transition to an amount proportional to their polarizability. We choose the Z axis to lie along the most polarizable direction in the chromophoric group. The second most polarizable direction, perpendicular to the Z axis, will be chosen as the Y axis, and the X axis will be chosen along the least polarizable direction, such that the axes X, Y, and Z form a right-handed coordinate system. The perturbing term $Axyz$ will be made up of contributions from all groups off of the principal polarizability axes. The contribution to the term $Axyz$ for a vicinal group will be negative if the potential on the chromophoric electron is lower in the direction of the vicinal group than it would be if the group were replaced in such a way as to introduce a plane of symmetry in the chromophoric group. Such a vicinal group will be more polarizable than the group which would make the chromophore symmetric, or else will carry a greater net positive charge. If the vicinal group has a net negative charge, its contribution to $Axyz$ will be positive, corresponding to a high potential for the chromophoric electron to move in that direction.

We consider next the transition to the lowest energy level of the chromophore. When the lowest transition is electric dipole allowed, the dipole lies along the Z direction. We can consider the transition to be from a state characterized by the harmonic oscillator in its ground state with quantum numbers, $n_1 = n_2 = n_3 = 0$, i.e., the state (0, 0, 0), to the state (0, 0, 1) with an excitation in the Z direction. The rotation in this case is given by

$$\phi = \frac{Nhe^2A}{6(2\pi)^3\mu^3\lambda^2}\left(\frac{n^2+2}{3}\right)\times \left(\frac{1}{\nu_2}-\frac{1}{\nu_1}\right)\frac{1}{(\nu_1+\nu_2)^2-\nu_2^2}\times\frac{1}{\nu_3^2-\nu^2}. \quad (8)$$

For this electric dipole transition in which the force constants have been chosen so that $\nu_1 > \nu_2 > \nu_3$, the rotation on the low frequency side of the absorption band follows the sign of the parameter A. When the lowest energy for a transition is magnetic dipole allowed, the transition is from a state in which the electron is predominantly in one direction to a state where the electron lies in another direction. From the manner in which we have chosen our axes, this will usually correspond to a transition from a state in the Y direction, with quantum numbers (0, 1, 0), to a state in the X direction, with quantum numbers (1, 0, 0). The transition energy, in terms of frequency, will be $\nu_1 - \nu_2$. The rotation is given by

$$\phi = \frac{Nhe^2A}{6(2\pi)^3\mu^3\lambda^2}\left(\frac{n^2+2}{3}\right)\times \left(\frac{1}{\nu_1}-\frac{1}{\nu_2}\right)\frac{1}{\nu_3^2-(\nu_1-\nu_2)^2}\frac{1}{(\nu_1-\nu_2)^2-\nu^2} \quad (9)$$

For this magnetic dipole allowed transition, the rotation on the visible side of the absorption again follows the sign of the parameter A. To determine the rotation, one must fit the potential of equation (6) to the chromophoric group and add the contributions to A from the groups which do not lie on the planes defined by the principal axes of the chromophore. The rotation will follow the sign of A.

As an example, we consider the contribution to the optical rotation of substituted cyclohexanones from the carbonyl transition which occurs at about 2900 Å wavelength. Figure 3(a) shows a cyclohexanone ring and Figure 3(b) shows the ring as it would appear when viewed from the oxygen along the C=O axis. The figure is drawn assuming that all angles are tetrahedral. The deviations which exist in the molecule will affect the positions of the substituents only slightly and will not affect the discussion. We consider the cyclohexanone skeleton locked in this configuration by side groups, as in bicyclic systems. Ring conformations will have to be determined when more than one conformation is possible. The Z direction is directed from the oxygen toward the adjacent carbon. The next most polarizable direction, the Y direction, is perendicular to Z and is in the plane containing the oxygen atom, C_1, C_{R2} and C_{L2} and is directed toward C_{R2}. The X direction will then point toward the top of the page as indicated in Figure 3(b). The transition is magnetic-dipole-allowed in the carbonyl group and is from a nonbonding state, primarily *2py* on the oxygen atom to the anti-bonding molecular orbital, π_x^*, located on the oxygen and carbon atoms. We can see that this satisfies the above conditions.

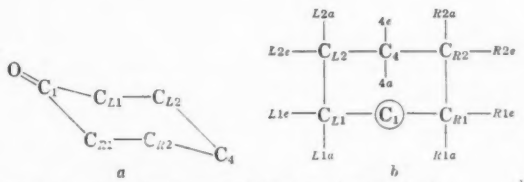

Figure 3 (e and a denote equatorial and axial substituents).

Consider next the contribution to the optical rotation which arises when substituents are placed at various equatorial and axial positions. It is convenient to assume that these positions initially contained hydrogen. The contribution to the parameter A will then arise from the difference in properties between the substituted group and the hydrogen. A group substituted at *R2e* will lie approximately on the plane defined by the Y and Z axes and will not contribute to the rotation. A group substituted at *R2a* will lie in the quadrant characterized by the coordinate signs, $X = -$, $Y = +$, $Z = +$, so that the product of the coordinate signs is negative. When the polarizability of the substituted group is larger than the polarizability of hydrogen, as in the case of a bromine, chlorine, or methyl group, the potential will be smaller in that octant and the sign of $Axyz$ will be negative. For these substituents at *R2a* the term A will be positive and the contribution to the rotation by this group will be positive. For the case of a substituted fluorine, since its polarizability is smaller than that of hydrogen, the term $Axyz$ must be positive in that quadrant and hence the value of A must be negative. A similar analysis of the other positions around the cyclohexanone ring

leads to the following table for the contribution to the rotation in cases where the substituent is more polarizable than hydrogen, such as bromine, chlorine, or methyl. For substituents which are less polarizable than hydrogen, such as fluorine, the values in the table for the sign of contribution to A will be of opposite sign.

Table 1

Position	Substituent	x	y	z	Sign of contribution to A
R2	*a*	−	+	+	+
	e	0	+	+	0
R3	*a* & *e*	+	+	+	−
4	*a* & *e*	+	0	+	0
L3	*a* & *e*	+	−	+	+
L2	*a*	−	−	+	−
	e	0	−	+	0

The magnitude of the contribution to the rotation will be approximately proportional to the difference in polarizability between the substituent and hydrogen. These findings provide the basis for the "octant rule" which was proposed by Djerassi [(*16*), page 178] and co-workers on the basis of a large number of rotatory dispersion measurements taken on substituted steroids.

Another example of this method is provided by lactic acid. The chromophore is the carboxyl group, and its rotation may be expected to determine the rotation in the visible range of the spectrum. If the oxygen atom of the carboxyl group is hydrogen-bonded to the hydroxide as shown in Figure 4, the Z axis will lie along the >C=O bond, chosen positive in the direction of the carbon atom. The second most polarizable axis, Y, will lie in the plane of the ring, chosen positive in the direction of the hydroxyl hydrogen. The X axis then points out of the plane of the paper. The most polarizable group lying out of the plane of the ring is the methyl group, which is in the octant, $X = +$, $Y = +$, $Z = +$. Since $Axyz$ must be negative, the rotation will be negative as it is known to be for D-lactic acid.

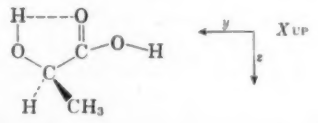

Figure 4. D-Lactic Acid.

The optical rotation for many molecules may be dependent on the internal conformation of the molecule. The harmonic oscillator model provides a rapid method of analyzing this situation. An example of the rotation of a molecule changing with internal conformation is provided by diethyl tartrate. This molecule may exist in two intramolecularly hydrogen-bonded forms as shown in Figure 5. Figure 5(*a*) shows a five-membered ring and Figure 5(*b*) shows a six-membered ring. The structures formed by using the carboxyl group at the other end of the molecule are similar to the ones shown. The chromophore is the $\diagdown C{=}O \diagup$ group, as in lactic acid, and we choose our axes in the carbonyl group as before. In the five-membered ring, Figure 5(*a*), the principal group out of the plane is the $-CHOHCO_2C_2H_5$, which lies in the quadrant $X = +$, $Y = +$, $Z = +$. Since $Axyz$ must be negative, A must be negative and the rotation will be negative for this structure. In the six-membered ring structure, Figure 5(*b*), the principal group out of the plane of the ring is the hydroxyl group which lies in the octant $X = -$, $Y = +$, $Z = +$; therefore, A will be positive and the rotation positive for this structure. The data for the optical rotation of diethyl tartrate indicate that these two forms may exist in solution (*20*), different forms being stable in different solvents. As the temperature increases, the rotation in all solvents tends toward a constant value, to be identified with the rotation of the non-hydrogen-bonded form. The temperature dependence of the rotation may be expressed as a sum of rotations of the various forms. Each of the hydrogen-bonded forms is in equilibrium with the non-hydrogen bonded form, the equilibrium constant being different in various solvents. This may be expressed as

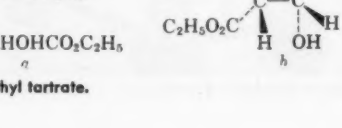

Figure 5. Diethyl tartrate.

$$\phi = M_5N_5 + M_6N_6 + M_nN_n$$

and

$$N_5 = N_ne^{-\Delta H_5/RT+\Delta S_5/R}; \quad N_6 = N_ne^{-\Delta H_6/RT+\Delta S_6/R}$$

where M_5 and M_6 are the molar rotations of the five- and six-membered rings respectively, and M_n the molar rotation of the non-hydrogen-bonded structure. N_i is the mole fracture of the ith species and ΔH_i and ΔS_i are constants for a particular solvent and the ith species. The observed values can be represented by choosing suitable values for ΔH and ΔS. Figure 6 shows as dotted lines two of the experimental curves; the corresponding calculated curves are shown as solid lines. The curves 1 are for the solvent alpha-nitro naphthalene and were calculated using $\Delta H_6 = 3$ *k* cal; $\Delta S_6 = 9$ eu;

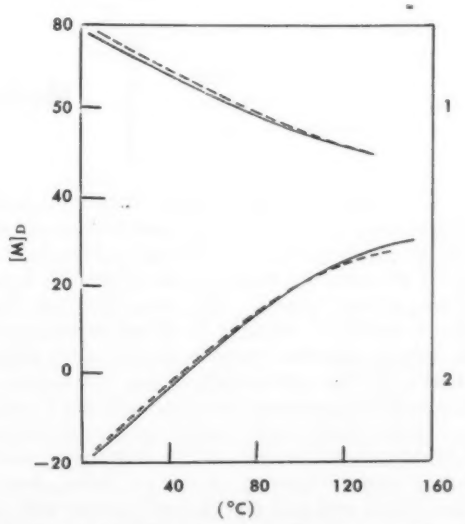

Figure 6. Solvent and temperature effects on diethyl tartrate. 1. α nitro napthalene. 2. Anti benzaldoxime.

$M_6 = 100°$. The curves 2 are for anti-benzaldoxime as solvent and the values $\Delta H_5 = 5.7$ kcal; $\Delta S_5 = 18$ eu; $M_5 = -40°$. The curves were calculated assuming only the five- or six-membered ring was present with the non-hydrogen bonded form, for which the value $M_n = 40°$ was used in both cases. These values for ΔH and ΔS are in the range of values which would be expected for hydrogen bonds.

This harmonic oscillator model can also be used to predict the absolute configuration of complex ions. As an example we will consider the optically active tris ethylenediamine cobalt(III) ion, $[Co(en)_3]^{+3}$, which is shown in one of its optically active forms in Figure 7, where the heavy lines represent the ethylenediamine groups. The most polarizable direction will be along the N—Co—N direction, chosen positive toward the top of the page. Either of the other two directions would have been equally good; however, their contribution to the rotation will be the same as the one given and their inclusion only multiplies the magnitude by a factor of three. The second most polarizable direction perpendicular to Z is directed up from the paper along the most polarizable component of the substituted groups. The X direction will then be as indicated in Figure 7. The groups which contribute

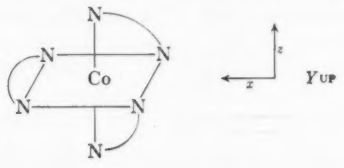

Figure 7.

to A will be the ethylenediamine groups lying in the octants $X = -, Y = -, Z = +$; and $X = -, Y = +, Z = -$. For both of these, since the product $Axyz$ must be negative, the value of A will be negative and the rotation will be negative. This is in agreement with the X-ray determination of the absolute configuration by Saito (*18*). A similar discussion will show that the same sign of rotation will be obtained when one of the ethylenediamine groups is removed and two other groups, such as chlorines, are put in its place.

It should be clear from our examples that the harmonic oscillator model for optical rotation provides a well founded, general method for predicting the relation between absolute configuration and conformation and the sign of the observed rotation.

We wish to thank the NSF for a research grant and a Cooperative Graduate Fellowship for one of the authors (L.L.J.).

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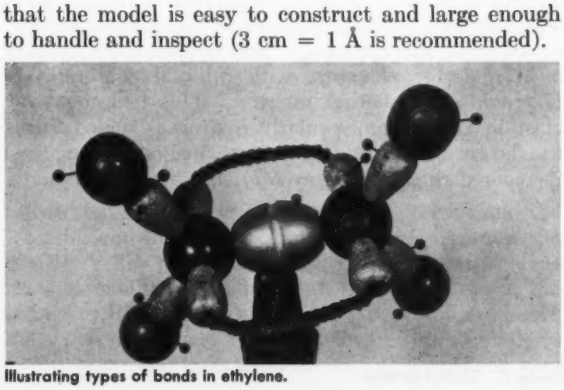
Models Illustrating Types of Orbitals and Bonding

A beginning student finds the concepts of orbitals, especially hybridized and bonded orbitals, difficult to comprehend. Models help, but the usual system of showing the situation after bonding is complete hides all inner detail. The accompanying photograph of a model of ethylene is offered as an example of the clearer viewing possible if the outer orbitals are shown in the unreacted shape. Materials are common and inexpensive: wooden balls for C and H atoms; ovals (small corks sanded to shape) for the p and hybridized sp^2 orbitals of C; maptacks indicate electrons; pipe cleaners show pi bond overlap. Wooden dowels and glue give desired rigidity and durability. The usual colors contrast different structures. Angles are easily made exact; the scale should be such that the model is easy to construct and large enough to handle and inspect (3 cm = 1 Å is recommended).



Illustrating types of bonds in ethylene.

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A Qualitative Elemental Analysis System Correlated with the Periodic Table

This article describes a new system of qualitative analysis. It has been developed especially for instructional purposes because of the belief that a course based on such a system offers unique potentialities as a means for teaching descriptive inorganic chemistry and the principles of chemical reactions.

One of these potentialities results from the opportunity which a system of qualitative analysis presents to systematize and correlate effectively a large and useful background of factual inorganic chemistry. This is especially true because the basis of qualitative systems is a process of first utilizing the more general properties of the elements for major group separations, thereafter proceeding to less general properties for the separation of subgroups, and, finally, to specific properties for individual separations and confirmatory tests. Unfortunately, this broad systemization is often lost by premature and undue emphasis on detailed material.

The effectiveness of qualitative analysis in this systemization will be determined largely by the extent to which the major groups of the system can be correlated with the fundamental properties of the elements, and therefore with their electronic structures and positions in the periodic table. This consideration has been primarily responsible for the development of this new system of analysis.

Groups Based on Periodic Relationships

During World War II there was developed at the California Institute of Technology for use by the Chemical Corps of the United States Army a system of elemental analysis which provided for the detection and estimation, by volumetric or colorimetric methods, of some 32 elements. Because this system was intended for organic as well as inorganic materials, the initial step consisted of a fusion of the sample with sodium peroxide and sucrose in a Parr bomb. When the fusion product was treated with water, a residue was obtained which consisted of those elements forming oxides or carbonates sufficiently insoluble and basic in character to remain as precipitates in the strongly alkaline solution. This residue was separated and called the *Basic Element Group*. The solution was divided into two portions. One portion was analyzed for those elements forming predominantly amphoteric oxides; these elements were called the *Amphoteric Element Group*. The other portion was analyzed for those elements forming predominantly acidic oxides and these elements were called the *Acidic Element Group*.

Contribution No. 2725 from the Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena.

These major groups were then separated into subgroups and these subgroups analyzed by (in most cases) modifications of conventional methods. This system was extensively tested and subsequently used by the field laboratories of the Chemical Corps during the war for the analysis of a wide variety of material. However, except for a brief description by Swift and Niemann in *Analytical Chemistry*, **26,** 538 (1954), the material has not been published.

The initial division of the elements into the three groups mentioned above offers an excellent opportunity to correlate the elements of these groups with the metallic, metalloid, and non-metallic elements. In addition, correlating the properties of these elements with their electronic structures and positions in the periodic table appears to offer distinct instructional advantages over the major group separations used in conventional systems of qualitative analysis. Also, the inclusion of the acidic elements (that is, the acid-forming elements) into a general system seemed advantageous when compared with the conventional treatment of the so-called cations by one system and the anions by another, which often seems an unsystematic appendage. Therefore, a simplified form of this system, modified for instructional use, was developed at the California Institute of Technology and has been used in preliminary form here and at UCLA for eight years; tabular outlines showing the group separations of this simplified system are presented in this article.

Since this is a system of elemental analysis, and since the major group separations differ so widely from conventional systems, there seemed little justification for adhering to the conventional list of "common elements." Rather, the elements included have been selected primarily because their properties and reactions illustrate the principles of typical chemical reactions, or of structural and periodic relationships. The number of elements included has been limited to those which could be covered in a one-semester course with two 3-hr laboratory periods per week; certain common elements not included are used as reagents and their properties discussed. Where less laboratory time is available certain elements and subgroups can be eliminated from the required work.

Qualitative analysis is justified in the undergraduate curriculum only if it is effective as a device to teach the principles and relationships stated above, yet experience has shown that the interest of the students is stimulated by the fact that this system has been derived from one which was developed to meet a professional need. Partly for this reason, the quantitative features of the Chemical Corps system have been retained and very simple and rapid methods for estimating the elements

have been provided. These estimations involve visual comparisons of color intensities, titrations with calibrated droppers, or in many cases simple comparisons of precipitant sizes; they can be eliminated altogether from courses having limited laboratory time. Even when this is done their availability is evidence to the student of the effectiveness of the separations and of the necessity of some degree of quantitative information for any valid interpretation of analytical results. Where time is available these estimations enable the student to evaluate his own capabilities; in addition, they can be used to teach the quantitative and stoichiometric nature of chemical reactions as well as the elementary principles underlying quantitative colorimetric and volumetric measurements.

The scale of operations adopted for this system represents a compromise between conventional macro and semimicro systems, and is a result of an effort to retain the advantages of each of these fields. Most of the operations involve solution volumes of 3–6 ml and can be performed in 15-ml test tubes or 25-ml conical flasks. Provision is made for the use of both centrifugation and filtration, depending upon the nature of the precipitate or subsequent operation. Simple equipment for suction filtration has been devised which in many cases is more rapid to use than centrifugation; use of such equipment minimizes congestion around centrifuges. It is believed that this scale of operation approaches the economy in reagent and equipment cost of the semimicro system, without the loss of quantitative significance which is characteristic of many such systems. The authors believe that this quantitative factor contributes greatly to the interest, challenge, and instructional potentialities of this qualitative system.

Group Separations

The group separations of this system of elemental analysis are shown in the accompanying tables.

Table 1 shows the treatment which has been substituted for the original fusion. A nickel crucible is used instead of the expensive Parr bomb, and a flux of sodium hydroxide, sodium carbonate, and sodium nitrate is used in place of the sodium peroxide-sucrose mixture. Effective fusions can be obtained by means of a low flame from an ordinary gas burner. Several thousand fusions have been made to date by freshman students without any accidents causing personal injury. Sodium peroxide was first used in the flux and was analytically effective; it was abandoned because of difficulties encountered in student use of this reactive and hygroscopic compound. The separation of the basic elements from the amphoteric elements obtained after a fusion with sodium peroxide and sucrose (or with sodium hydroxide and either sodium peroxide or sodium nitrate) is surprisingly superior analytically to that obtained by the conventional aqueous treatment with sodium hydroxide and peroxide. This effect is attributed to the precipitate being much less voluminous and less hydrous as a result of the fusion.

At Cal Tech and at UCLA no fusions are done until after analyses of solution unknowns of single subgroups have been made. However, lecture and class discussions of the fusion process emphasize the structural and periodic relationships responsible for the behavior of the elements in this treatment; these discussions of the fusion also point out that chemical reactions can take place in other than aqueous solutions and that there are many similarities, as well as differences, between reactions in aqueous and fused media. Instead of attempting to cover in these lectures the wide variety of compounds and reactions involved in the specific separations and confirmatory tests, the emphasis is on general knowledge and understanding of the oxide and hydroxyl compounds of the elements and their reactions. Subsequent discussions treat the sulfur compounds and their reactions, and their similarities to those of oxygen. The general principles, especially those relating to bond types, which are presented in connection with the oxygen and sulfur compounds then can be extended to other types of compounds. In the laboratory, even though the student begins his analysis with a solution of a subgroup, he is required to write equations for the reactions which each element of that subgroup undergoes in the fusion and subsequent subgroup separations.

Analysis of Basic Element Group

Table 2 shows the methods used for the separating of the elements of the *Basic Element Group* into subgroups. Emphasis is continued on the behavior of oxide and hydroxyl compounds. The hydrolysis and precipitation of the Titanium Group elements illustrate the

Table 1. Behavior of the Elements after Fusion of the Sample and Treatment of the Fusion Product with Water. Separation of the Basic Element Group

Fuse the sample in a nickel crucible with NaOH, $NaNO_3$, and Na_2CO_3. Treat the melt with hot water.

Residue	*Solution*	
Basic Element Group	*Amphoteric Element Group*	*Acidic Element Group*
Fe_2O_3, MnO_2, TiO_2	$Pb(OH)_4^{-2}$, $Cu(OH)_3^{-}$[c]	I^-, Br^-, Cl^-
$BaCO_3$, $CaCO_3$, $Mg(OH)_2$	AsO_4^{-3},[d] $Sn(OH)_6^{-2}$	PO_4^{-3}, AsO_4^{-3},[d] SO_4^{-2}, F^-
Ag_2O, Ag, CuO, NiO	$Al(OH)_4^-$, $Zn(OH)_3^-$	
[PbO_2, SnO_2][a]	CrO_4^{-2}, VO_3^-	NO_3^-, CO_3^{-2}[e]
Separate sample	One half of the solution is used for the analysis of the amphoteric elements and the other half for the acidic elements.	
Soluble Basic Element Group		
Na^+, K^+[b]		

[a] Elements in brackets appear where indicated only when present in large quantities or as the result of imperfect separations.
[b] A separate sample is analyzed for sodium and potassium, the Soluble Basic Element Group, because their hydroxides and carbonates are soluble, and because sodium salts are used in the fusion.
[c] Only a small amount of copper is dissolved and appears here.
[d] Arsenic will occur with both the Amphoteric and Acidic Element Groups and may be detected in either group.
[e] Separate samples are analyzed for nitrogen and carbonate carbon.

Table 2. Separation of the Basic Element Group into Subgroups

Treat the fusion residue with HNO_3 and $NaClO_3$.
Precipitate: MnO_2, [PbO_2, SnO_2]. *Gas:* Cl_2, ClO_2.
Solution: TiO^{+2}, Fe^{+3}, Ba^{+2}, Ca^{+2}, Mg^{+2}, Ag^+, Cu^{+2}, Ni^{+2}.
Neutralize. Add CH_3COOH and CH_3COONH_4. Heat.

Precipitate:	*Solution*
Fe_2O_3, $CH_3COOFeO$, MnO_2, TiO_2 [PbO_2, SnO_2]	Ba^{+2}, Ca^{+2}, Mg^{+2}, Ag^+, Cu^{+2}, Ni^{+2}, H^+, CH_3COOH, CH_3COO^-, NH_4^+, NO_3^- Add excess NH_4OH and $(NH_4)_2CO_3$.
The Titanium Group	*Precipitate* $BaCO_3$, $CaCO_3$, $MgCO_3$ The Alkaline Earth Group
	Solution $Ag(NH_3)_2^+$, $Cu(NH_3)_4^{+2}$, $Ni(NH_3)_4^{+2}$; OH^-, NH_4OH, NH_4^+, CH_3COO^-, CO_3^{-2}, NO_3^- Add NaOH. Boil out NH_3.
	Precipitate Ag_2O, CuO, $Ni(OH)_2$ The Metal Ammine Group
	Solution Discard

general effect of oxidation state on the tendency of an element to form covalent bonds with oxide or hydroxyl ions. The separation of the Alkaline Earth Group from the Metal Ammine Group shows the effect of complex ion formation on the solubility of certain hydroxides and the differing tendencies toward complex ion formation of the transition elements as compared with those of the Alkali and Alkaline Earth families.

Analysis of the Amphoteric Element Group

One portion of the fusion solution is analyzed for the *Amphoteric Element Group* elements and Table 3 shows the separation of these elements into two subgroups by

Table 3. Separation of the Amphoteric Element Group into Subgroups

Neutralize one half of the fusion solution with HCl; make 0.4 *F* in HNO_3.
Pb^{+2}, Cu^{+2}, H_3AsO_4, H_2SnCl_6, Al^{+3}, Zn^{+2}, Cr^{+3}, $V(OH)_2^{+2}$, (also Na^+, K^+, and the acidic elements).
Add CH_3CSNH_2. Heat. Add $(NH_4)_2SO_4$ (to buffer at 0.07 *M* H^+). Again heat.

Precipitate	*Solution*
PbS, CuS, As_2S_3, SnS_2, S The Sulfide Group	Al^{+3}, Zn^{+2}, Cr^{+3}, $V(OH)_2^{+2}$ (CH_3CSNH_2, CH_3CONH_2, H^+, Cl^-, and acidic elements) The Aluminum-Chromium Group

the precipitation of the Sulfide Group. No attempt was made in developing this system to completely eliminate the use of sulfide separations. The importance of sulfide reactions, analytically and generally, made such an attempt seem unrealistic. Lectures can be used to point out the adjacent positions of oxygen and sulfur in the periodic system and to stress the analogies between the various oxygen and sulfur compounds. The similarity in the types of reactions used in oxide and sulfide separations is used to reinforce the previous treatment of oxide chemistry.

Thioacetamide is used instead of gaseous hydrogen sulfide to precipitate the Sulfide Group. The two types of reactions involved in precipitating metal sulfides by thioacetamide are discussed; the process serves as an example of precipitations from homogeneous solutions. The modifications of procedure and changes in hydrogen ion concentrations required by the substitution of this precipitant for gaseous hydrogen sulfide are explained.

The Sulfide Group is separated into subgroups by means of an alkaline sulfide solution, as shown in Table 4. This shows again the analogy between the sulfide

Table 4. Analysis of the Sulfide Group; Separation of the Lead Group from the Arsenic Group

Treat the Sulfide Group precipitate with NaHS and NaOH

Residue	*Solution*
CuS, PbS The Lead Group	AsS_4^{-3}, SnS_3^{-2}, S_2^{-2} Acidify with HCl As_2S_3, SnS_2 (H_2S, S) The Arsenic Group

and oxide (or hydroxyl) anions of certain elements.

The precipitation of the Aluminum-Chromium Group elements and their separation into subgroups are shown in Table 5. These procedures again illustrate the effects of oxidation state on the properties of and types of bonds formed by the elements.

Table 5. Precipitation of the Aluminum-Chromium Group and Separation of the Aluminum Group from the Chromium Group

The Aluminum-Chromium Group solution: Add NH_4OH (to *p*H of 7–8). Heat.
Precipitate: $Al(OH)_3$, $Cr(OH)_3$, [$V(OH)_4$, VS_2, ZnS].
Add NaHS.
Precipitate: ZnS.

Precipitate: Aluminum-Chromium Group (as above)	*Solution:* VO_3^-, VS_4^{-3} (orange color)
Dissolve in HNO_3. Evaporate.	
Solution: Al^{+3}, Cr^{+3}, $V(OH)_4^+$, Zn^{+2}, H^+	CH_3CSNH_2, CH_3CONH_2, HS^-, Cl^-.
Dilute. Add 10 *F* H_2O_2 (Al^{+3}, Cr^{+3}, Zn^{+2}) HVO_4 (orange color). Add excess NaOH. Heat.	Match color with known V solution. (Only small quantities of V will be here.)
Solution: $Al(OH)_4^-$, $Zn(OH)_4^{-2}$, VO_3^-, OH^-, CrO_4^{-2} (yellow color)	
Saturate with CO_2.	

Precipitate	*Solution*
$Al(OH)_3$, $ZnCO_3$ The Aluminum Group	CrO_4^{-2}, VO_3^-, HCO_3^-, H_2CO_3 The Chromium Group

Acidic Element Group

The second portion of the fusion solution is analyzed for the *Acidic Element Group* elements and Table 6 shows, first, the preliminary separation of silicon and the more typically amphoteric elements from the more acidic elements; these elements are then separated into groups. This first procedure again illustrates the application of *p*H control by a buffer system to effect a desired separation. The separation of the Halogen

Table 6. Separation of the Acidic Element Group into Subgroups

Saturate the other half of the fusion solution with CO_2.

Precipitate: H_2SiO_3 [also $SnO_2 \cdot (H_2O)_x$, $PbCO_3$ $CuCO_3$, $Al(OH)_3$, $ZnCO_3$] Heat with H_2SO_4. Add NaCl and H_2O. *Precipitate:* $SiO_2 \cdot (H_2O)_x$ (presence of silicon) *Solution:* $SnCl_6^{-2}$, $PbCl_4^{-2}$, $CuCl^+$, Al^{+3}, $ZnCl_4^{-2}$. Discard.	*Solution* I^-, Br^-, Cl^-; HPO_4^{-2}, $HAsO_4^{-2}$ (CrO_4^{-2}, VO_3^-);[a] SO_4^{-2}, F^-; NO_3^-, NO_2^-; HCO_3^-, Alkali elements. Add HNO_3 and $AgNO_3$.		
	Precipitate AgI AgBr AgCl The Halogen Group	*Solution* H_3PO_4, H_3AsO_4, $Cr_2O_7^{-2}$, $V(OH)_4^+$; HSO_4^-, HF_2^-, Ag^+, H^+ Neutralize with Na_2CO_3. Add CH_3COOH and CH_3COONH_4	
		Precipitate Ag_3PO_4, Ag_3AsO_4 (Ag_2CrO_4, $AgVO_3$)[a] The Phosphorus Group	*Solution* HSO_4^-, HF_2^-, NO_3^-, Ag^+, CH_3COOH Analyze for sulfur and fluorine. Analyze separate samples for nitrogen and carbon.

[a] The analysis for Cr and V is made in the Amphoteric Element Group; they precipitate with As and P in this group.

from the Phosphorus Group and the subsequent precipitation of the Phosphorus Group show the relative effects which the hydrogen ion concentration can exert on the solubility of the salts of strong and weak, especially polyprotic, acids.

The methods involved in the analysis of the various subgroups of the system are mostly conventional in nature. Preference has been given to methods involving typical reactions and yielding general information, rather than to extremely sensitive spot tests with complicated organic compounds. The analysis for the Titanium Group elements (Table 2) departs from the usual sequential pattern in that individual separations are not made, but the detection and estimation of each of the three elements is made colorimetrically on separate portions of the group solution. These methods involve the red ferric thiocyanate complex, the permanganate ion, and the orange titanium (IV)-peroxy compound.

Justification for a New Qualitative System

Because of the continuously expanding boundaries of chemistry, the justification for a qualitative analysis course in the undergraduate curriculum has been repeatedly and properly questioned. When a biological organism ceases to grow degeneration begins. This principle can be applied to a scientific field, where growth can be measured by the research productivity and resulting progress of that field. On this basis there has been an outstanding lack of growth in the field of qualitative analysis in the past few decades. As stated previously, the authors are convinced of the value of qualitative analysis as in instructional tool. Therefore, they believe that the time and extensive amount of experimental effort involved in developing this system will have been warranted if it leads others to reexamine the justification for undergraduate courses in qualitative analysis—especially if this reexamination leads to renewed interest in developing more effective systems and courses of qualitative analysis.

Acknowledgment

Most of the experimental work involved in modifying the original system developed for the Chemical Corps to this present instructional system was carried out by Professor Eliot A. Butler, now at Brigham Young University. The analytical effectiveness of this system is in large part due to his work. We are indebted to the faculty and students at UCLA and Cal Tech for many helpful suggestions resulting from class use of this system. Investigations supported by the National Science Foundation have led to the successful use of thioacetamide as a sulfide precipitant and to an understanding of the reactions involved.

Suggestions for Binding the Journal of Chemical Education

The Editor has corresponded with several readers who have expressed concern over the problems of binding the current volume of THIS JOURNAL. They point out that much valuable material is lost from the permanent files of libraries by the discard of all pages numbered "A000." We share this concern and suggest the following alternatives.

(1) Bind the complete issues. The advertising pages contain valuable information about instruments and books which is worth a fraction of an inch of shelf space. The Book Exhibit list, for example, which appeared in September, pages A646—A701, is one of the handiest ready references in print.

(2) Indicate pages selected for discard by clipping the corners. Binderies will follow instructions, even to assembling into one section the Tested Demonstrations, Chemical Projects, etc. Remember that cover photographs illustrate feature articles.

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Determination of Saponification Equivalents of Phenolic Esters

Although a variety of methods for determining the saponification equivalents of esters of carboxylic acids are described in the texts and literature of organic analysis,[4–6] these procedures give uniformly unsatisfactory results when applied to esters of phenols. The object of this paper is to show that by making a few key changes in existing techniques, a procedure is evolved which is not only applicable to ordinary alkyl esters of carboxylic acids, but gives excellent results with a wide variety of esters of phenol and other aromatic hydroxyl compounds as well.

Procedure

Reagents. A hydrolysis solvent which consists of approximately 0.25 *N* NaOH in 50% by volume *p*-dioxane is prepared by dissolving the appropriate quantity of reagent grade NaOH pellets in a quantity of distilled water and diluting to twice that volume with purified *p*-dioxane. If this hydrolysis solvent is kept in tightly capped polyethylene bottles it is very stable and requires only occasional restandardization.

The *p*-dioxane is prepared by refluxing any high quality *p*-dioxane, such as Eastman No. 2144 or Fisher "Spectro-Grade," over excess metallic sodium until the metal is bright, and then distilling off the *p*-dioxane as required.

Approximately 0.25 *N* HCl is prepared by diluting reagent grade concentrated HCl with distilled water. The HCl solution is standardized against tris(hydroxymethyl)aminomethane[7] using methyl orange indicator, and then the hydrolysis solvent is standardized by determining the acid-base ratio using phenol red indicator. 0.04% phenol red in water (Eastman A 541) is suitable for use here and wherever phenol red indicator is called for. The normalities of the solutions must be determined to within ±0.0001 for most accurate results.

To determine the saponification equivalent of an ester, a sample of approximately 0.4 g is weighed to 0.1 mg in a 125-ml ₮24/40 Erlenmeyer. 25.00 ml of the standardized hydrolysis solvent is pipetted into the flask and the flask is fitted with a 30 cm ₮ 24/40 Liebig condenser capped with a drying tube containing Ascarite CO_2 absorbant. (The ground glass joint should have a light coating of silicone grease to insure an airtight seal.) The contents of the flask are refluxed for ½ hour or longer if required. At the end of this time the heat is removed and the condenser is rinsed into the flask with distilled water. 20.00 ml of the standardized HCl solution is pipetted into the flask and the appropriate indicator is added.

If during reflux the solution has remained colorless, 5 drops of phenol red indicator are added. If the solution has developed an intense coloration during reflux, 10–15 drops of thymolphthalein indicator are added instead. (Eastman A 1091, 0.5% in alcohol is suitable.)

Immediately after addition of the indicator the flask is stoppered and cooled by swirling under the tap. The separation of free crystalline acid may occasionally be observed at this point. It may be ignored. Additional 0.25 *N* NaOH in 50% *p*-dioxane is now added from a buret until the end point is reached.

If phenol red was added, the color change is from yellow to rose at the end point. If thymolphthalein was added to a strongly colored solution, the color change at the end point of this same titration will depend upon the color of the particular solution. If the solution is colorless, thymolphthalein will change to deep blue at the end point. The end point color changes for thymolphthalein in the presence of several colored phenolate ions are shown in Table 2. With either indicator the color change is very sharp, usually within one drop. No blank correction is required if the procedure outlined above is followed carefully.

The saponification equivalent of the ester is calculated from the usual equation

$$S.E. = \frac{W \cdot 1000}{N_B \cdot V_B - N_A V_A} \qquad (1)$$

where

$S.E.$ = saponification equivalent,
W = ester sample weight (gm.),
N_B = normality of NaOH in 50% *p*-dioxane,
V_B = *total* volume of NaOH in 50% *p*-dioxane used (ml),
N_A = normality of HCl solution, and
V_A = volume of HCl solution used (usually 20.00 ml).

Hydrolysis Solvents

Most commonly used saponification procedures make use of an alcoholic or glycolic solution of NaOH or KOH for the hydrolysis solvent. When solutions of this type, prepared as ordinarily recommended (that is, without prior purification and distillation of the organic compound) are allowed to stand on the shelf, rapid discoloration and change in titer almost invariably occur. Even freshly prepared solutions of this type will deteriorate rapidly if refluxed. Although the above

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[4] Cheronis, N. D., and Entrikin, J. B., "Semimicro Qualitative Organic Analysis," 2nd ed., Interscience Publishers, Inc., New York, 1957, p. 735 and references therein.

[5] McElvain, S. H., "The Characterization of Organic Compounds," rev. ed., The Macmillan Co., New York, 1956, p. 133.

[6] Shriner, R. L., Fuson, R. C., and Curtin, D. Y., "The Systematic Identification of Organic Compounds," 4th ed., John Wiley & Sons, Inc., New York, 1957, p. 235.

[7] Whitehead, T. H., J. Chem. Educ., 36, 297 (1959).

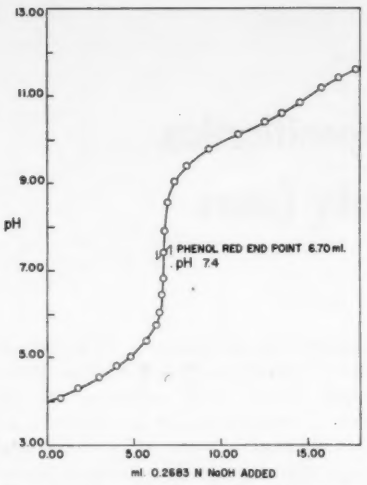

Figure 1. Back titration pH curve for phenyl benzoate. 0.4666 gm phenyl benzoate refluxed in 25.00 ml 0.2683 *N* NaOH in 50% (v/v) *p*-dioxane-water and quenched with 20.00 ml 0.3089 *N* HCl; 5 drops phenol red indicator added. *S.E.* = 200.5.

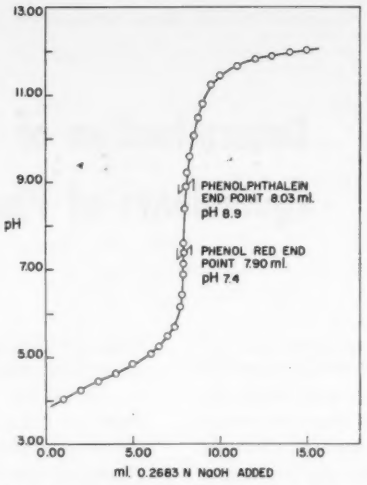

Figure 2. Back titration pH curve for ethyl benzoate. 0.4081 gm ethyl benzoate refluxed in 25.00 ml 0.2683 *N* NaOH in 50% (v/v) *p*-dioxane-water and quenched with 20.00 ml 0.3089 *N* HCl; 2 drops phenol red indicator added. At phenol red end point 5 drops phenolphthalein indicator added and titration continued. *S.E.* = 154.1.

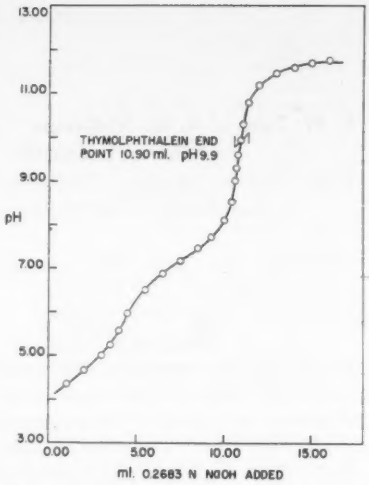

Figure 3. Back titration pH curve for *p*-nitrophenyl benzoate. 0.4142 gm *p*-nitrophenyl benzoate refluxed in 25.00 ml 0.2683 *N* NaOH in 50% (v/v) *p*-dioxane-water and quenched with 20.00 ml 0.3089 *N* HCl; 10 drops thymolphthalein indicator added. *S.E.* = 119.9 *S.E.* × 2 = 239.8.

difficulties are minimized when the alcohol or glycol is carefully purified, others remain. In the presence of a phenolic ester the purified hydrolysis solvents still tend to discolor during reflux, obscuring the end points of subsequent titrations. It may well be that the liberated phenol rather than the solvent itself is decomposed. Even in the absence of ester, an easily observable blank reaction occurs, presumably due to reaction between base and the flask itself. Whatever the cause, the experimental results obtained with the above solvents usually leave something to be desired.

Proceeding on the assumption that the difficulties mentioned might be eliminated if the organic component of the hydrolysis solvent contained no reactive or ionizable group, *p*-dioxane, a cyclic di-ether, was tried. The results were excellent. No detectable blank reaction occurs and, except when the phenol liberated during hydrolysis is strongly colored, the solutions remain absolutely colorless. *p*-Dioxane has other advantages. It is a good solvent for water insoluble esters, particularly at elevated temperatures. It is miscible with water in all proportions and in mixtures with water maintains a boiling point near 100°. This is helpful in promoting rapid saponification at reflux. It is readily available, and is easily purified in one step. It should be emphasized that *p*-dioxane ordinarily contains peroxides which, if not removed by prior distillation of the *p*-dioxane from metallic sodium, will consume base during the saponification process and cause inaccurate results.

End Point Indicators

Phenolphthalein, the usual end point indicator for saponification equivalent titrations, is not suitable for phenolic esters. The factors which lead to the selection of phenol red and thymolphthalein instead are clearly shown in Figures 1–3.

Figure 1 shows the back titration *p*H curve obtained during a determination of the saponification equivalent of phenyl benzoate, a typical carboxylic acid ester of a phenol.[8] The vertical portion of the curve extends from *p*H 6.5 to *p*H 8.5, and occurs after all the 0.25 *N* HCl used to quench the reaction and all the benzoic acid (*p*K 4.7) liberated during the hydrolysis have been neutralized. Above *p*H 8.5 the curve slopes markedly due to the beginning of the neutralization of the phenol (*p*K 10.0) present in the medium. The visual end point for phenolphthalein (*p*H 8.9) is not located on the vertical portion of the titration curve and consequently the usually sharp color change is smeared out. In the presence of a phenol, therefore, phenolphthalein is not satisfactory as an end point indicator. However, Figure 1 shows that phenol red is suitable since its visual end point (*p*H 7.4) is located on the vertical portion of the curve. Bromthymol blue, neutral red, and *m*-nitrophenol also have suitable end points. However, phenol red was deemed best since the brilliant acidic (yellow) and basic (violet) colors provide a striking color change at the end point.

Figure 2 shows that the change in end point indicator from phenolphthalein to phenol red does not limit the usefulness of the technique to phenolic esters only. The back titration *p*H curve in Figure 2 was obtained during a determination of the saponification equivalent of ethyl benzoate, a typical carboxylic acid ester of an aliphatic alcohol. The vertical portion of the curve, which extends from *p*H 6.5 to *p*H 8.9, includes the visual end points for both phenolphthalein (*p*H 8.9) and phenol red (*p*H 7.4).

Unfortunately, phenol red is not suitable for all phenolic esters. Phenol itself (*p*K 10.0) lowers the upper limit of the vertical portion of the titration curve in Figure 1 from 8.5 to 8.0. Any aromatic hydroxyl compound with a *p*K of 9.2 or less lowers this upper

[8] All *p*H measurements made at 25°C with Beckman Model G *p*H meter, No. 39170 reference electrode, and No. 40498 glass electrode standardized at *p*H 7.00.

limit sufficiently to obliterate the sharp phenol red end point as well. The back titration *p*H curve shown in Figure 3 illustrates this point. It was obtained during a determination of the saponification of *p*-nitrophenyl benzoate. The phenol red end point is completely obliterated. This is because the neutralization of the *p*-nitrophenol (*p*K 7.2) liberated during the hydrolysis is underway before the neutralization of the benzoic acid is complete. To complicate matters, the *p*-nitrophenol and its anion impart an intense yellow color to the solution.

Figure 3 shows, however, that a new, nearly vertical portion of the titration curve occurs between *p*H 9.2 and *p*H 10.6. This vertical rise occurs after the aromatic hydroxyl compound has also been neutralized. Fortunately, thymolphthalein has a visual end point (*p*H 9.9) in this zone, and is an excellent end point indicator for the titration. In this particular instance the observed end point color change is from yellow to green, as indicated in Table 2, due to the presence of the yellow *p*-nitrophenolate ion. No other common indicator was found having either the proper color change interval or a strong enough color change to permit its use in this application.

The decision of when to use thymolphthalein rather than phenol red is simplified by the fact that *most* aromatic hydroxyl compounds with *p*K's below 9.2 owe their increased acidity to the presence of a nitro substituent on the aromatic nucleus which invariably renders the compound and its anion strongly colored in solution. As indicated earlier, thymolphthalein is added as the end point indicator when the saponification solution becomes strongly colored during reflux. More than the usual quantity of indicator is called for in order to guarantee a strong color change.

There are, of course, a number of aromatic hydroxyl compounds with *p*K's below 9.2 which do not impart a visible color change to the hydrolysis medium, e.g., *o*-halogenated and polyhalogenated phenols. When an ester containing such a moiety is encountered, such as *o*-chlorophenyl benzoate, phenol red fails to give a sharp end point even though the solution is colorless, and the resulting saponification equivalent is inaccurate. However, if the saponification is repeated and thymolphthalein is used as the titration indicator, a sharp end point is obtained and the calulated saponification equivalent, as shown in Table 2, is satisfactory.

In order to eliminate the occasional necessity of repeating saponification, an attempt was made to develop a mixed indicator containing both phenol red and thymolphthalein which would be useful for all esters. Unsatisfactory results were obtained.

Table 1. Comparison of Known Molecular Weights with Saponification Equivalents Determined Using Phenol Red Indicator

Ester	Molecular weight	*S.E.*	Per Cent difference
Ethyl acetate	88.1	88.6	+0.57
Ethyl benzoate	150.2	149.9	−0.20
Methyl phenylacetate	150.2	151.6	+0.93
Methyl salicylate	152.2	152.0	−0.13
Phenyl salicylate	214.2	212.5	−0.79
1-Naphthyl acetate	186.2	185.5	−0.38
2-Naphthyl benzoate	248.7	247.8	−0.36
Phenyl benzoate	198.2	198.7	+0.25
Phenyl-*p*-methoxybenzoate	228.2	227.5	−0.31
Phenyl-*p*-chlorobenzoate	232.7	232.4	−0.13
Phenyl-*m*-nitrobenzoate	243.2	240.7	−1.03
p-Methoxyphenyl benzoate	228.2	230.5	+1.01
p-Methoxyphenyl-*p*-methoxybenzoate	258.3	258.3	0.00
p-Methoxyphenyl-*p*-chlorobenzoate	262.7	263.0	+0.11
p-Methoxyphenyl-*m*-nitrobenzoate	273.2	272.5	−0.26
p-Chlorophenyl benzoate	232.7	231.7	−0.43
p-Chlorophenyl-*p*-methoxybenzoate	262.7	262.2	−0.19
p-Chlorophenyl-*p*-chlorobenzoate	267.1	267.6	+0.19
p-Chlorophenyl-*m*-nitrobenzoate	277.7	275.3	−0.86
p-Methylphenyl benzoate	212.1	212.2	+0.05

Results

Tables 1 and 2 compare the saponification equivalents of 26 esters determined using the procedure outlined with known molecular weight values. As may be seen, the method gives accurate results with carboxylic acid esters of both aromatic and aliphatic hydroxyl compounds. The average deviations of the results from the theoretical values are well within ±1%. With the exception of the first three esters listed in Table 1, previously reported procedures would fail to give satisfactory saponification equivalents.

The calculated saponification equivalents of the phenolic esters of monocarboxylic acids shown in Table 2 have been multiplied by two for comparison with the known molecular weights. This is because equation (1) is derived on the assumption that each mole of ester hydrolyzed consumes one mole of base. At the thymolphthalein end point both the carboxylic acid and phenol are neutralized, so that each mole of ester consumes two moles of base.

There are two types of esters which cannot be successfully analyzed using the technique outlined in this article. The saponification equivalents of carboxylic acid esters of polyhydroxyl aromatic compounds cannot be determined due to the ready destruction of the polyhydroxyl aromatic ring under basic conditions. Esters of *p*-hydroxybenzoic acid and *p*-hydroxycinnamic acid also fail to give meaningful results due to the ready decarboxylation of these acids under basic conditions.

This work was supported by a Frederick Gardner Cottrell grant from the Research Corporation and one from the West Virginia Wesleyan College Research Fund.

Table 2. Comparison of Known Molecular Weights with Saponification Equivalents Determined Using Thymolphthalein Indicator

Ester	Molecular Weight	*S.E.* × 2	Per Cent difference	End point color change
o-Chlorophenyl benzoate	232.7	234.2	+0.64	colorless-blue
m-Nitrophenyl benzoate	243.2	243.2	0.00	orange-brown
m-Nitrophenyl-*p*-methoxybenzoate	273.2	273.0	−0.07	orange-brown
m-Nitrophenyl-*p*-chlorobenzoate	277.7	278.6	+0.32	orange-brown
m-Nitrophenyl-*m*-nitrobenzoate	288.2	291.4	+1.11	orange-brown
p-Nitrophenyl benzoate	243.2	243.0	−0.08	yellow-green

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The Liebig-Pasteur Controversy

Vitality without vitalism

> I think ... that the disputes between Mechanists and Vitalists are unsatisfactory .... One is never quite sure what is meant by "Mechanism" and by "Vitalism"; and one suspects that both names cover a multitude of theories which the protagonists have never distinguished and put clearly before themselves.[1]

The legacy of 19th century science includes not only sweeping advances in all major areas, but also heated controversies. This was the first century of "modern" science, of systematic experimentation by professional scientists. It was also the first century of "modern" disputes, in which careful, but selective, experimentation was added to age-old stubbornness. Famous is the debate between Liebig and Pasteur concerning the nature of fermentation and a general explanation of what are now called enzymatic reactions. For many years, this was a living issue and the arguments were vigorous and often bitter. Liebig gave full rein to his sarcastic, inventive mind against the solid Gallic logic of Pasteur.

Today, fermentation and related processes are believed to be due to the action of particular chemical substances: enzymes. Their relation to inorganic catalysts has been established in principle. Many of the theoretical concepts applied to catalysis in living systems are derived from studies on strictly chemical systems. Even a further serious question concerning the nature of enzymes has been resolved: they are now almost universally recognized as belonging to the class of proteins, either pure or combined with known structures. That is, they are members of a larger category of chemical compounds whose properties and behavior have been studied for many decades. It is perhaps worth mentioning that general agreement on this last point has only been reached since 1930. The ensuing 30 years have not been sufficient to dispel completely the air of mystery which surrounds discussions concerning enzymes, especially (but not exclusively) those in the nontechnical literature.

Indeed, in some sense the controversy lives on in two distinct although overlapping forms. On the one hand, historians of science and biographers of the protagonists prolong the debate in their accounts of 19th century events as well as in some of their interpretations of modern views concerning enzymes. On the other hand, the Liebig-Pasteur controversy can be considered as one example of the continuing unsolved mechanism-vitalism problem which dates back to the pre-Socratics. It is worth while examining the evidence in view of both current interests. Careful consideration can help to set the record straight: it can help to scrape bare the positions actually held by Liebig and Pasteur from the accumulated encrustations of almost 100 years of partisan commentary. Further, a new presentation can shed light on the kinds of arguments and opinions which are put forward by mechanists and vitalists.

Historical

In 1839 Liebig published a paper in his *Annalen* (*1*) summarizing his views on fermentation, decay, and putrefaction. The extensive analyses which had been carried out in his laboratory afforded him ample evidence of the similarities between organic and inorganic matter. This view was supplemented by his own researches on natural products which had led to the isolation from bitter almonds of "emulsin," an obviously nonliving material which catalyzed the decomposition of amygdalin (*2*). Finally, Liebig had at his disposal Berzelius' recent discussion on "catalysis" (*3*) in which the latter made no attempt to distinguish inorganic and organic (in the wider sense) catalysts by any fundamental difference in composition or mode of action.

Liebig's theory compared fermentation and related phenomena to other decomposition processes. He assigned violent motion to the particles of the catalyst as it decomposed and assumed that the transmission of this motion to the reactants brought about their decomposition in turn. In modern terminology, catalysts were "energy rich" substances which could transfer their (translational) energy to certain chemically similar reactant molecules and enable them to undergo decomposition. The emphasis was on analogy to processes known from inorganic chemistry. He repeatedly compared the action of heat and acids to that of ferments. In several cases he pointed out the specificity of certain catalysts and tried to relate this to structural similarities between the activating compound and its "substrate." The theory accounted for catalyst specificity and neatly related observations in inorganic chemistry, organic chemistry, and general physiology.

In August of 1857 Pasteur presented a paper before the faculty of science at Lille and repeated it three months later before the Académie des Sciences (*4*). He reported that his observations on the fermentation of sour milk to produce lactic acid led him "to a completely different point of view" concerning fermentation. Subsequent papers on other forms of fermentation, as well as his concurrent interest in *génération dites*

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Contribution No. 2730 from the Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena.

[1] Broad, C. D., "The Mind and Its Place in Nature," Routledge and Kegan Paul Ltd., London, 1925.

spontanées, supported his original conclusions. In every case the chemical change observed was accompanied by the appearance and multiplication of large numbers of microscopic "globules." These appeared to be living cells, and were also components of the catalyst introduced to start the reaction. If they were destroyed by heat or removed by filtration, the fermentation process ceased. Also, various types of fermentation led to the production of optically active products; a fact, which, to Pasteur, indicated the involvement of living organisms. These observations sharply distinguished fermentation processes from other catalytic phenomena and suggested that the chemical changes brought about during fermentation were "correlative" with the growth of the globules, or bacteria. In his most extensive publication on the subject (*5*), Pasteur fit his theory into the general framework of cellular biological concepts and paid tribute to scientists who had previously suggested similar explanations, among them Cagnard de la Tour, Schwann, and Turpin.

These contradictory views provided the groundwork for the controversy. On the surface the sides seem clear. Pasteur is a vitalist; he claims that living organisms are necessary for the fermentation process. Liebig is a mechanist; he explains all catalysis, including fermentation, by a chemical-kinetic, mechanistic theory. For Liebig, Pasteur's bacteria were only an incidental event accompanying the breakdown of products and catalysts.

Who was right? There is general agreement today that enzyme action does not require vitalistic explanations; and, after Buchner's demonstration of extracellular, alcoholic fermentation in a yeast extract (*6*), the last doubts concerning the requirement of viable cells were dispelled. The answer appears clear-cut.

Yet, it all depends on which commentator you read. We may dismiss those biographies of Pasteur and Liebig which are by their partisans. There is no doubt for Vallery-Radot (*7*) in writing about his father-in-law nor for Volhard (*8*) writing about his teacher and benefactor! However, even Singer (*9*) concludes a brief discussion of this topic with the statement that, "... unlike Liebig, Pasteur *realized* that fermentation demanded the presence of living organisms" (*9*). This is hard to reconcile with Conant's terse conclusion, "Pasteur's generalization was wrong, but it was marvelously fruitful" (*10*). The issue is hardly clarified when one remembers that Liebig was actually a vitalist who publicly answered some of the ardent mechanists of his day.

Vitalism

Some of the confusion described above results from the same causes that led to the original extended debates. An attempt to defend one champion against charges from the other side easily leads to exaggeration and sometimes to misstatements. However, by far the greatest problem arises from a poor understanding of the word "vitalism" and its application to biological and chemical theories.

The Oxford English Dictionary defines "vitalism" as "the doctrine that the origin and phenomena of life are due to or produced by a vital principle, as distinct from purely chemical or physical force." In similarly defining the word Webster's points out that it is opposed to mechanism. The meaning of vitalism is clear: it refers to a theory, or a group of theories, which explains some or all manifestations of living organisms in terms of something other than the laws of chemistry and physics. Vitalism provides some form of explanation in non-mechanistic terms.

Since the "laws of chemistry and physics" and even the laws of mechanics are subject to change without notice, it is sometimes a little difficult to pin down the boundaries between mechanism and vitalism. C. D. Broad argues (*11*) that the only "pure" mechanistic explanation is one which elucidates all phenomena in terms of a single kind of stuff, which undergoes a single kind of fundamental change, which can be explained in terms of a single elementary causal law and which has only a single and simple principle of composition. This is rather limiting and makes just about every tenable theory a vitalistic one by default, a situation much to Broad's liking.

However, such fine points need not bother us here. It is enough to recognize that any mechanistic theory says that the laws of chemistry and physics, or the fundamental laws of physics, since today scientists agree that chemistry is a subdivision of one branch of physics, are enough to explain the behavior of living things. A particular vitalistic statement or a particular vitalistic argument is one which explains a phenomenon characteristic of a living thing in terms of something other than the laws of physics, something not reducible to a law of physics.

Although fairly obvious, it is important to note that it is not necessary to assume that all properties of living things require an explanation in terms of vital forces in order to hold a vitalistic position. On the contrary, it is only necessary to say that a single such property is not explainable in terms of the physical laws. Indeed, no vitalist would be likely to take the inclusive extreme position. If a mouse is dropped, it falls just as a stone does. I doubt that anyone has ever felt compelled to supply separate explanations for these observations on living and non-living matter. If a bat is substituted for the mouse, the result is quite different. Yet, even here, no sensible person nowadays would suggest that the flying creature violates the laws of gravity. In fact it is doubtful that for the last several centuries anyone would have suggested that the flight of the bat, or of any other winged creature, required anything but a mechanistic explanation. I am speaking here only of the act of flight, not of possible reasons why certain animals have the organs which enable them to fly.

What, then, is the essence of vitalism? Just how does it differ from mechanism? This depends on the view that is held; vitalistic theories vary widely.

Hospers (*12*) considers four ways of stating the issues between mechanism and vitalism. These involve discussions concerning a divine Creator of life, a non-material life force, emergence, and non-reducibility. The last of these is fundamental. At the minimum, any vitalistic position holds that the basic laws governing the behavior of living organisms are not reducible to the laws of chemistry and physics. Non-reducibility is a necessary condition for vitalism. Unlike the first three positions, an argument supporting only non-reducibility need not state explicitly how the laws of biology differ from the laws of physics. Since the

"laws of biology" are not only subject to change but are as yet uncertain, this problem becomes extremely complex. Among others, many modern physicists interested in biology have struggled with it. For example, Delbrück (*13*) argues that it is possible that the fundamental laws of biology may be related to the fundamental laws of modern physics as the latter are related to the laws of classical physics. He draws an analogy between the present state of biology and the condition of physics in 1910, and suggests that the resolution of some impasses between various approaches to molecular biology may prove to require similarly drastic revisions of concepts as were suggested by the non-classical explanations offered as resolutions for problems in atomic physics half a century ago. An important part of any such argument is to say not only that we do not know the fundamental laws of biology, but also that a new conceptual framework may be required to establish sound laws. If a biologist accepts this suggestion and acts accordingly, he could be satisfied with explanations of biological phenomena which are not couched in the same terms as the present laws of physics, although they may be similar to them. I shall call such a position a minimum vitalistic position.

The first three issues listed above all have in common the introduction of some particular force or agent into explanations concerning living things, or, in the case of emergence, at least the introduction of purpose. Further, they all readily lead to positions which place complete explanations of the phenomena in question outside the realm of physical science. A vitalistic explanation based on one of these positions would be characterized by its reference to the particular alternative to mechanism chosen; it would be recognized by its invocation of a divine being, an *élan vital* or *entelechy*, or an emergent property such as purpose, as the cause of some phenomenon. In any case, it would be held that the phenomenon in question could not be fully understood by reference to the laws of physics, and also that one of the alternative postulates would provide an adequate explanation. Although emergence theories (those that hold that the whole is greater than the sum of its parts) are often couched in vague terms involving tendencies or the mysteries of life, on the whole all the above, which I shall call positive vitalistic positions, are readily recognized by their reference to some non-spatial and usually non-empirical principle.

The above brief outline of possible vitalistic positions emphasizes one important point: it is not reference to living things or to living processes which makes one a vitalist. If this were the case, every biologist would necessarily be a vitalist. Only the explanation of a phenomenon of life or of a process associated with living things by means other than the laws of chemistry and physics represents a vitalistic point of view. The minimum vitalistic position offers a particular explanation to account for the purported particular behavior of living things. The vitalism-mechanism issue is concerned with causes and origins.

Liebig's View

Since Liebig's theory tried to explain all catalytic processes including fermentation by the same theory involving mechanical motion and chemical interaction, it is clearly not vitalistic. He even went further and first denied the existence of living cells in fermenting mixtures; later, when forced to concede their reality, he at least denied that they were causally related to the fermentation process.

Any attempt to reconcile this view with Liebig's vitalistic sentiments fails. Indeed, this phase of Liebig's life is difficult to integrate with his scientific work. One reason we revere Liebig today is because he extended chemistry to include a wide range of topics which had previously been within the preserve of biologists, often vitalists. With supreme confidence, Liebig approached problems in agriculture and physiology as strictly chemical tasks. In his sweeping researches in these areas he never had any doubts that the materials he isolated from living things were chemical substances, amenable to chemical study and subject to the laws of inorganic chemistry. His popular discourses in opposition to what he called "excessive-mechanism" must be attributed to other than scientific ways of thought.

Pasteur's View

There is no doubt that Pasteur considered fermentation to be intimately connected with living cells. However, this is not enough to make his position a vitalistic one. To recognize (or assume) that some property or function is found only in living objects is simply to classify it, not to explain it. The question of causation still remains.

Further, it is important to distinguish between those phenomena which are part of the life process and those which are only found with it. Members of the former class are sometimes difficult to define, since the question, "what is life?" is involved here. However, members of the latter class are readily recognized. For example, no reasonable person would contend that the ability to fly is necessary for life to manifest itself. Yet only living things are capable of voluntary flight.

For Pasteur, fermentation was a process carried out only by viable cells during their growth and multiplication. He clearly recognized the metabolic significance of fermentation, that the products observed were remnants of the substrate after it had been utilized by the cell. However, he chose his words carefully; over and over in the many papers on this subject he repeats the same phrase, "*les fermentations sont correlatives d'un acte vital.*" This is a statement of the same kind as "only living things are capable of flight." Certainly it does not in any way imply a vitalistic hypothesis. In fact, it says nothing about the cause of fermentation or about the laws which govern living things.

Pasteur was a careful man. He stayed close to the facts, his facts. His experimental observations were that fermentations were chemical processes carried out only by living cells during their life cycle, and the experiments permitted nothing further. In fact, the papers entitled "*Sur la nature de la fermentation*" are disappointing in the sense that on this question they contain little more than reiterations that fermentation is correlative with the development of the cells. His repeated publications show forcefully and determinedly that living cells carry out the fermentation process, that they multiply and grow during it, and that there can be no fermentaion in their absence. Again and again, he admitted that he was ignorant of the true

cause of fermentation. Probably his statement that "fermentation, properly so-called," required living cells was motivated more by a desire to restrict the term to experimentally similar situations than by an attempt to save his theory even at the risk of a circular argument. Also, without our wider framework of reference, it was impossible for Pasteur to realize that all his researches on fermentation, involving different bacterial species and leading to a variety of products, are only a small class of enzyme reactions. After working for 20 years and publishing more than 50 papers and notes, it was reasonable that Pasteur considered that his work encompassed the field of what we now call enzyme chemistry and that the known unorganized ferments (that is, extracellular enzymes) were peripheral examples not worthy of classification under the same heading.

One of the experimental results which seemed of particular importance to Pasteur was that fermentation could produce optically active compounds. Pasteur believed that only living organisms were capable of producing asymmetrical products from optically inactive starting materials. Since fermentation could bring this about, it must be related to a life process. This argument was crucial for Pasteur; it may have influenced his unshaking faith in the correctness of his theory, which was somewhat of a speculation when first advanced and certainly contrary to the most generally held view as exemplified by Liebig's paper. It may be significant that Pasteur's first contribution on the subject concerned itself with lactic acid fermentation, which produces an optically active product, rather than with the more common vinous fermentation, which produces the simpler inactive products alcohol and carbon dioxide. We can use Pasteur's interest in this point to trace his thinking concerning causes. Molecular asymmetry was something which Pasteur felt he understood very well and he speculated about it much more than he speculated on the nature of fermentation.

In examining his views on optical activity and molecular asymmetry we may first ask, how did the asymmetric molecules produced by fermentation arise? Pasteur was explicit on this point (*14*): the formation of asymmetric molecules must arise because the bacteria themselves contain asymmetric molecules. He considered that the chemical action of the ferment on its substrate involves the formation of a diastereoisomeric pair, only one member of which is capable of reaction. He clearly drew the direct analogy to the resolution of a racemic mixture of acids by an optically active base. Thus, the chemical action of yeast on its substrate involves the same principles as a chemical separation of inorganic optically active compounds. Note that nothing but the laws of chemistry is invoked. More than that, something about the nature of the living ferment is inferred by analogy to a non-living system.

However, Pasteur did not stop here. What is the origin of the asymmetry found only in nature, the asymmetric molecules produced only by living organisms? Pasteur believed that this was caused by strictly natural forces, forces known to physics. He commented that the earth is asymmetric because of its axial rotation and that this might provide a natural source of asymmetry. Pasteur felt that the existence of asymmetric force fields was a reasonable cause for the production of asymmetric molecules; he even considered this a subject worthy of experimental work. He performed crystallizations in the presence of strong magnetic fields, expecting to demonstrate a kind of asymmetric induction. He hoped to grow plants in an environment where, by the use of mirrors, they were subject to the influence of an "inverted" sun that rose in the west and set in the east, thus producing plants containing compounds of "unnatural" configuration.

Neither the reasonableness of his theory nor the practicality of the experiments need concern us here. The only important point is that Pasteur felt that asymmetry was intimately related with living systems and yet he sought mechanistic causes for it! In this instance where he went beyond the facts and sought a fundamental explanation for a phenomenon of life, he turned immediately to physics. No alternative kind of interpretation was even suggested.

To summarize, Pasteur's theory of fermentation and related processes is not in any way vitalistic. He did not invoke positive vitalistic causes; he did not even take a minimum vitalistic position. His contribution to the controversy involved the report and reiteration of experimental results and not the proposing of causes. Where he did seek causes he looked for them within the known laws of chemistry and physics.

Critique of Commentators

Despite the apparently forthright and clear stand Pasteur took, he is persistently dubbed a vitalist. It might be argued that the word "vitalism" is used synonymously with "vitality" to mean simply "involving life." Remarkably enough, this is often not the case. Those who call Pasteur a vitalist use the term correctly, although this does not detract from its erroneous application in this instance. A good example of this sort of argument is found in the influential biography of Pasteur by René Dubos (*15*). In introducing Pasteur's views on fermentation (p. 119) he wrote,

> The 20th-century scientist does not know how to introduce life into his equilibrium reactions any better than the 19th-century chemist knew where to place yeast in the formula of alcoholic fermentation. However, so great were the triumphs of physicochemical science during the "wonderful century" that many scientists had enough confidence, or perhaps merely enough intellectual conceit, to ignore the difficulty, and to refuse to recognize the existence of an unsolved mystery in fermentation and putrefaction. Pasteur was willing to introduce mystery in the problem by stating that yeast was a living being and fermentation an attribute of its life.

A few pages later, in describing Pasteur's antagonists, he wrote (p. 123),

> In the kingdom of science, there were probably many who did not share the official optimism, and who did not believe that the time had come when everything could be accounted for in terms of known physicochemical forces. But the priests of the new faith—Berzelius, Liebig, Wöhler, Helmholz, Berthelot and others—were the supreme rulers of scientific thinking... the new prophets had pronounced anathema on anyone who preached the doctrine of vitalism.

As those who had previously supported the vitalistic theory of fermentation and putrefaction he listed Cagnard de la Tour, Schwann, Kutzing, and Turpin.

These passages clearly indicate that Dubos thinks

of vitalism as the doctrine which opposes mechanism. They also suggest a leaning in favor of the vitalistic view of nature over the mechanistic one. However, this is not important to us except as perhaps supplying a motive for his entire discussion. What is more important is that the classification given puts people in the wrong camps! I have already pointed out that Pasteur's views on this question are not consistent with a vitalistic position. Similar arguments could be presented for Schwann. Although generally recognized as one of the founders of the cellular theory, he was an ardent and vocal mechanist. And these positions are by no means contradictory. The cellular theory makes a statement about a property of living matter: that it is always organized in complex units designated cells. It does not say anything about the origin of these units or the means by which their behavior may be explained. As a matter of fact, Schwann thought that cells could be precipitated out of solutions containing only inorganic materials, if all the right ingredients were present in the proper proportions. It is no mere coincidence that a generally available reprint of excerpts from Schwann's monograph on the cell theory (*16*) begins with a discussion of vitalistic (teleological) and mechanistic explanations of living matter and comes out firmly in favor of the latter view.

Contrariwise, Liebig definitely did not believe that "everything could be accounted for in terms of known physicochemical forces." I have already alluded to his vitalistic philosophy. A fuller discussion can be found in the comprehensive work by Metz (*17*). Even Whöler remained a vitalist, despite his reputation for having driven vital force out of organic chemistry. He too is misrepresented in Dubos' classification.

Curiously enough, Dubos referred at great length to Pasteur's fundamental mechanistic inclinations. However, he described them (p. 114) as the wild-eyed imaginings of a romantic mind.

> Pasteur carried with him into the grave the dream of his scientific youth—the fantastic vision of developing techniques for the creation or modification of life by introducing asymmetrical forces into chemical reactions.

A man who believes that life can be created or modified in this manner by the action of physical forces can hardly be labeled a vitalist.

The influence of Dubos' work can be illustrated by a recent article in THIS JOURNAL (*18*) which essentially summarizes his discussion:

> Turpin . . . Cagnard de la Tour, Schwann, and Kutzing were the unacknowledged (*sic*) forefathers of Pasteur's viewpoint. It was they who first eloquently expressed the sum and substance of Pasteur's dictum: The phenomena of alcoholic fermentation were inexplicable without resort to a vitalistic hypothesis.

Nature of the Controversy

If neither Pasteur nor Liebig held vitalistic positions, why was there a controversy? An answer is possible, although perhaps not one as dramatic as might be hoped. As is so often the case in modern science, Liebig and Pasteur carried out different experiments, arrived at different conclusions based on these experiments and on their general outlook, and stuck with them. Each one refused to recognize the other's references and analogies. This situation must be familiar to every practicing scientist.

Liebig speculated about fermentation mainly on the basis of other experiments. His evidence was drawn from the then known extracellular enzymes; therefore he had no reason to try to overcome a deepseated inclination to avoid considering living cells. Pasteur insisted on working only with enzymatic systems which, during his lifetime, could not be separated from living cells. He in turn ignored the work on digestive enzymes, which dated back to the previous century.

It does not seem desirable to dwell for long on this short-sightedness exhibited by two great scientists, whose main bodies of work are immortal. Certainly larger issues were involved. Liebig was opposed to the germ theory, while Pasteur became its most famous and ardent supporter.

It is easy for us to criticize the protagonists in the light of our present scientific beliefs and general climate of opinion. Liebig's refusal to recognize the microscopic evidence concerning the existence of cells strikes us as extremely short-sighted. But this attitude must be viewed in the framework of his background and theoretical views. Liebig was a student of Berzelius and Gay-Lussac; like them, what he explained, he explained in terms of chemistry and physics. If that was not enough, then he attempted no interpretation at all. Or to put it another way, if an explanation of a group of observations was possible, then that explanation had to be in terms of chemistry. In any case, new facts are often not enough to destroy a theory; a different frame of mind, a new way of looking at things, is required. And such a transformation of thought is most difficult for someone intimately involved in supporting one side of an issue. The same kind of criticism and excuse can be made for Pasteur. He became deeply committed to the cellular, germ theory and this dominated his thinking.

Finally, in order to make perfectly clear the kind of position taken by Pasteur, it is worth while to consider a truly vitalistic explanation of asymmetry. Pasteur noted the asymmetry found in living things and attributed it to the action of blind asymmetric forces. He further hoped to show experimentally that asymmetry could be created by the use of available asymmetric forces.

Here is another view, taken from a recent book concerned with fundamental causes of natural phenomena (*19*):

> It may be that life came on earth once, and once only, and that all life . . . has come from that one center. This seems suggested, if not proved, from the fact that certain chemical substances are known in two forms . . . known as dextro-rotatory and laevo-rotatory . . . if these substances be made synthetically in the laboratory of the chemist, they are almost invariably found to consist of equal quantities of either kind. If, on the other hand, they are prepared from the substances of living plants or animals, in which they occur naturally . . . they are found to be made up wholly of one type. This is by no means a conclusive argument—various ways of accounting for it may be imagined—but it does not by any means support the feeling, that there is an unholy and unending struggle between the creatures of the living world. It is, on the other hand, in general agreement with the evolutionary explanation, if it is thought that the whole of life comes from one original act, which may, in some sense, be identified with the religious idea of an act of creation by the Diety.

I shall not concern myself here with the validity or pertinence of this argument; what is important is its

form. No attempt is made to explain the experimental "fact." Instead, the observation is used to bolster a particular view concerning evolution, a view which, by its very nature, is not amenable to experimentation. This approach is perhaps of little interest to the scientist, but it certainly illustrates a marked contrast to Pasteur's attempts to resolve a similar question. The above is the view of a man who believes in a positive vitalistic view of life and who uses experimental evidence to support this view. Pasteur used experimental evidence to draw conclusions which were amenable to further experiment, all within the framework of the then known laws of physics.

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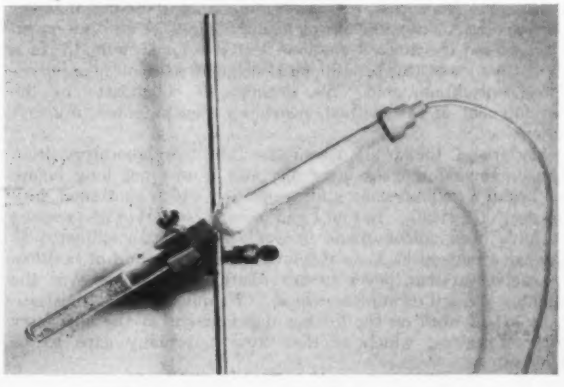
Adapting Hospital "Castoffs" for Use in a Chemistry Laboratory

Hospitals throughout the country dispose of hundreds, possibly thousands, of bottles daily. Among them are those which once held solutions which were given intravenously. When these bottles and their connections are to be disposed of, we requisition them for use in our chemistry laboratories. Those of volumes from 100 ml to 1000 ml are particularly useful for qualitative and quantitative analysis solutions. Besides being of convenient size they are also heavy-walled and have rough calibration markings on them. In our organic chemistry laboratory classes we have found use for the tubing connections, plastic or metal pinch clamps, and stoppers. The unusual rubber caps to which are attached narrow plastic tubing have been incorporated as an integral part of our "Aitchtuess" generators (see photograph). These adaptations have been a source of financial saving to the department, as well as a lesson in improvisation to the students.



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The Discovery of Germanin by Oskar Dressel and Richard Kothe

Both the human and the animal inhabitants of the tropical regions, such as India, South America, and (especially) Africa, are subject to the ravages due to trypanosomes—one-celled protozoan flagellates, larger than bacteria, which are transmitted through the bite of the bloodsucking tsetse flies. At first the trypanosomes live in the blood stream, then in the lymph channels, and finally in the cerebrospinal fluid. They produce remittent fever and swollen lymph nodes; later a general physical and mental weakness develops into the true "sleeping sickness," an anapathetic condition which advances with severe emaciation into somnia, loss of consciousness, and death. Unless treated early, death is almost certain. These pestilences are so prevalent in many areas that stock-raising has become impossible. However, a fundamental change in the treatment came with the introduction of the drug Germanin.

Ernest Fourneau (1872–1949), the leading French chemotherapist of his time, wrote in 1924:

> Toward the close of 1920 there was talk for the first time of a new remedy for trypanosomes under the name Bayer 205, which had been discovered in the laboratories of the dye manufacturers Friedric Bayer & Company. The first publications indicate that this substance is far more effective against trypanosomes than all of the other remedies which have previously been used . . . .It is an important discovery, which opens new and in fact unlooked for horizons.

From Dyes to Drugs

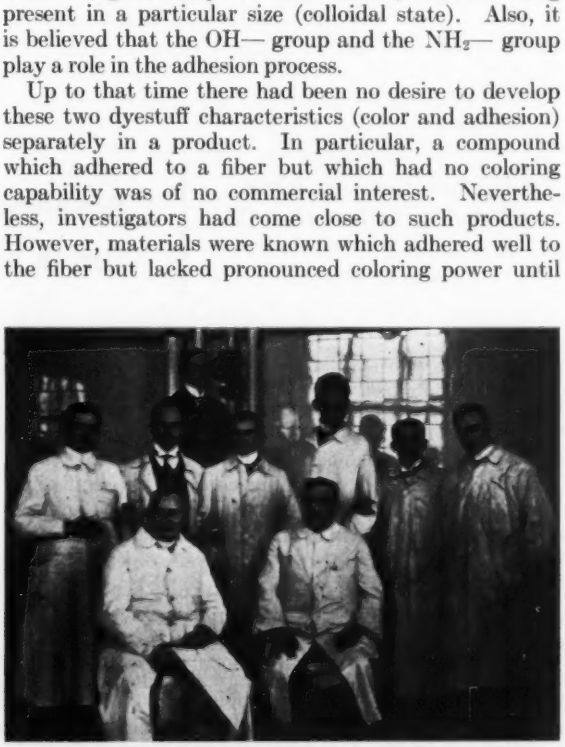
The drug was discovered by my father, Dr. Oskar Dressel, together with Dr. Richard Kothe. My father (1865–1941) had started to work in the scientific laboratory at Elberfeld in 1891. In his autobiographical notes he wrote:

> I quickly was immersed in the field to which I remained faithful for the entire next decade: the chemistry of naphthalene. I had come upon this field through a conversation with the eminent azo chemist Dr. Ulrich, who was complaining to me that many of the important sulfonic acids of naphthalene were still unknown. I considered the matter and soon searched for a method. In a short time I was able to astonish Ulrich with a sample of naphthalene-1,4-disulfonic acid. We decided to collaborate in the development of the method, which we then patented, my first patent.
>
> My friend Richard Kothe came into this laboratory from Leipsic several months after me, and it was not long before we began a collaboration and association which continued until his death (in 1926). In him I had found a co-worker who seemed to have been custom-made for me: scientifically adjusted to the same pattern as I, an indefatigable worker, and of faultless character, so that never in our relations was there ever the slightest discord or unpleasantness. We quickly and enthusiastically set to work on the further development of the chemistry of naphthalene, which at that time had many gaps to be filled in.

These joint labors, which resulted in numerous dyestuffs, continued until 1913. At that time, Mr. Heymann, director of the scientific laboratory, asked Dressel and Kothe to prepare several dyestuffs which had been ordered from the firm by two French researchers Mesnil and Nicolle. These dyes were later to be tested for their value in medicine. There was much interest in the work of Paul Ehrlich who had found that certain dyes had a toxic action on pathogenic organisms. He had placed high hopes on trypan red, which had been found in the laboratory to be extremely toxic against trypanosomes but had failed in veterinary trials in Africa.

Chemists learned by experience about the various actions of the functional groups and their position in the dye molecule. For example, the azo group —N=N— is responsible for the color of the azo dyes. For a long time it has been known that the ability of a dye to be extracted from aqueous solution and immediately to attach itself on the cotton fiber is due at least in part to an elongated shape of the molecule, or to its being present in a particular size (colloidal state). Also, it is believed that the OH— group and the NH_2— group play a role in the adhesion process.

Up to that time there had been no desire to develop these two dyestuff characteristics (color and adhesion) separately in a product. In particular, a compound which adhered to a fiber but which had no coloring capability was of no commercial interest. Nevertheless, investigators had come close to such products. However, materials were known which adhered well to the fiber but lacked pronounced coloring power until



Richard Kothe (left) and Oskar Dressel (right), seated with co-workers in the Laboratorium Elberfeld (1917).

they were chemically treated *in situ*. Some of these "developing dyes" were without any color at all in the first stage.

It was at this point that Dr. Roehl entered the picture. He had been trained under Paul Ehrlich and had been brought to Elberfeld to carry on chemotherapeutic studies on the products made by Bayer & Co. He sent out a general request:

> Please send me your dyestuffs so that I may test them on infected mice; but please send me only *colorless* dyes. Of what value is a drug, no matter how effective, if it gives my mice red or blue noses and ears and does the same later to the patients in the clinic?

Consequently, when Dressel and Kothe had prepared the materials ordered by Mesnil and Nicolle, they sent samples to Roehl. The materials were found to be ineffective, but this gesture of cooperation prompted Roehl to renew his request for "colorless" dyes. In addition, Dressel and Kothe decided to make an independent effort in this field. They selected molecules of a kind entirely different from those chosen by Mesnil and Nicolle; they decided to explore the field of urea chemistry. Four years of hard work followed, filled with uninterrupted series of trials. Their 15 years of experience in dyestuff chemistry profited the new line of endeavor. My father wrote:

> While I was away from the laboratory at Elberfeld to do some work at Leverkusen (both laboratories belonged to Bayer & Co.) Dr. Kothe made a very fine discovery in that he converted 2-amino-5-naphthol-7-sulfonic acid (J acid) by means of phosgene into a urea, which on coupling with diazo compounds yielded very valuable red wool dyes. The chain could be readily lengthened still more if amino-benzoyl nuclei were introduced, for instance *m*-aminobenzoyl J-acid, and converted into the corresponding urea by means of phosgene. We decided to make some efforts along this line.
>
> ... It soon was discovered that several compounds of this kind, particularly those with *four* aminobenzoyl groups, had a powerful though still inadequate effect on blood parasites. This finding of course encouraged us to make a still more intensive study of the matter than we had planned initially. Since our director Heymann was also pleased with the way things were developing, we enthusiastically embarked upon as comprehensive a study as possible of the large field.... The successes soon were greater and we were able to apply for the first patents. But not long thereafter World War I broke out. However, since we had already spent several years without achieving a really brilliant success in the chemotherapeutic field, our further efforts were regarded with some doubt by the supervising authorities, especially since we previously had had such excellent success in the dyestuff field. It must be noted that Mr. Heymann did not lose hope and therefore used his influence to secure permission for us to continue our experiments.
>
> We prepared more than 1000 ureas of the naphthalene series and had them tested. Finally in the autumn of 1917 we found the decisive compound: the urea obtained from *m*-aminobenzoyl-*m*-amino-*p*-toluyl-1-naphthylamine-4,6,8-trisulfonic acid. The action of this material on trypanosomes was outstandingly greater as compared with all the previous products.

The history of the application of this compound in the treatment of diseases caused by trypanosomes is best studied in papers and books put out by medical and veterinary writers. At present, Germanin is used in conjunction with other agents but it has not been replaced entirely. It appears on the market under a variety of names such as Suramin sodium, Bayer 205, Antrypol, Naganol, etc. Patent protection was withdrawn from the Germans after World War I and consequently the field was wide open to the many who entered the race to prepare modifications of the original compound. Probably more than 2500 similar compounds have been tested.

How a Discovery Is Made

In retrospect it will perhaps be of interest to review the path by which the discovery of Germanin was accomplished. The discoverers tilled the urea field because the compound produced with J acid and which gave the initial success belonged in this category. It was entirely logical for them to "choose the best horse in the stable." Secondly, its structure obviously was well suited to enlarging of the molecule. To a chemist who was well versed in both naphthalene and dyestuff chemistry, it was a natural step to include a naphthalene sulfonic acid, since such components serve to make large dye molecules soluble in water. The aminobenzoyl group likewise was a natural choice to an organic chemist, and the success of the rosanthrene dyes brought this group to the attention of the discoverers of Germanin. It must be emphasized that the compounds which they prepared for Mesnil and Nicolle played no particular role in the discovery, nor may any compound which might have been brought to their notice in the patent literature be regarded as the basis for the discovery.

There is no absolute rule which states how a molecule should be constituted if it is to possess particular chemotherapeutic activity. Success in this field can come only by combining a good knowledge of chemistry and chemotherapeutics with hard work and much patience. Discoveries such as Germanin do not arise because somebody has a lightning-like inspiration; such feats are legendary. Good fortune (luck) doubtless often plays a part but only when combined with competence in the field and hard work. However, hard work alone is never a guarantee of success. My father's notes on the discovery of Germanin closed appropriately with the lines from Goeth's "Faust," which in the Bayard Taylor translation read:

> Not Art and Science serve alone;
> Patience must in the work be shown.
> Long is the calm brain active in creation,
> Time, only, strengthens the fine fermentation.

✦ ✦ ✦

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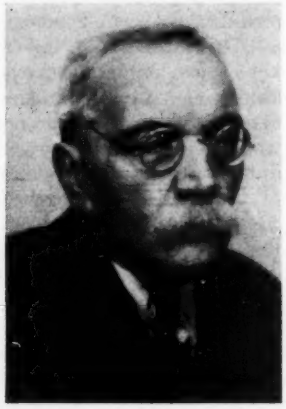
Aleksei Evgen'evich Chichibabin

Aleksei Evgen'evich Chichibabin was a member of the old guard which represented classical organic chemistry in Russia and which aided in the development of this science. He was one of the scientists who founded a pharmaceutical chemical industry in pre-revolutionary Russia. He aided in the industrialization of his country in the first World War and especially during the first Five-Year Plan in the USSR, which improved the position of the organic chemists and the technological teams in that country.



Aleksie Evgen'evich Chichibabin, shortly before his death in 1945.

Chichibabin was born in 1871 in the village of Kuzemino near Poltava in the Ukraine. At the age of 17 he received his general school certificate and entered the University of Moscow as a student in the Physico-Mathematical Faculty. He was forced to earn the money for his living and studies. He lived at the "Lyapinka," a home for penniless students, where their lives resembled those of the heroes of the Gorki novel "The Lower Depths" more than of ordinary students. In spite of all difficulties and misfortunes, he managed to graduate successfully at the age of 21. As a student he felt a strong desire for scientific work and in 1891 gave his first lecture at the Russian Chemical Society, "On the Action of Hydriodic Acid on Propylbenzene," but he was not allowed to carry on this work at the University. This was because his teacher, Professor Markovnikov, was suddenly discharged for political reasons, and the successor, Professor Zelinsky, did not wish to collaborate with the pupil of his predecessor. The young Chichibabin therefore willingly began to work with the assistant, Konovalov. During this period he earned his living writing articles for the newspapers such as the "Russian Gazette." He finally obtained a small position as chemist in the laboratory of the Union for the Progress of Industry. In order to obtain a background for experimental scientific work, he worked as a teacher in various secondary and professional schools. At length he became assistant to Professor Kablukov and in 1901 he was appointed Privat-Docent. His first lectures were given at the Agricultural Institute and concerned the experimental progress of organic chemistry.

In spite of the difficulties under which he lived and the hard working conditions, in 1903 he presented to the University of Moscow a dissertation "On the Products Obtained by the Reaction of Halogen Compounds on Pyridine and Quinoline." Professor Zelinsky gave a negative judgment on this work, and Professor Sabaneev ironically called Chichibabin "the self-educated man." Nevertheless the faculty approved the dissertation and granted him the degree of Master of Chemistry in 1904. The negative opinion of his work was based on the fact that he was working by himself instead of collaborating with the then recognized school of Russian chemists.

In 1905 he was chosen as Professor of Inorganic Chemistry in Warsaw, but he was soon forced to resign from this position because he could not agree with the working conditions and the spirit which prevailed at that time in the University of Warsaw. He therefore resumed his position in Moscow.

During 1908 he became Professor of Chemistry in the Polytechnic School in Moscow and from 1909 to 1929 he served, with some intervals, as Dean of the Faculty of Chemistry there. In 1912 he received his doctorate at the University of St. Petersburg (now Leningrad) with a dissertation "On Trivalent Carbon." In 1929 he was elected a regular member of the Academy of Sciences of the USSR and Director of the Scientific Institute for the Synthesis of Natural Compounds. For his activity he was awarded the Butlerov and Lenin Prizes, and a number of other distinctions.

According to the Russo-German commercial agreement of 1904, pre-revolutionary Russia was not allowed to refine coal tar. By this agreement, Germany had a monopoly for distribution of pharmaceutical products. It was therefore quite clear in the first days of World War I that Russia was short of medical supplies. For this reason Chichibabin established a "Committee for Organizing Medicament Production," and he became its first president. With his voluntary collaborators he worked out in detail methods for manufacturing synthetic medical preparations and he initiated their industrial production. He organized the collection of medical plants and arranged facilities for the extraction

Presented at the First Congress for Pure and Applied Chemistry of Yugoslavia, June, 1960, in Rijeka, Yugoslavia.

of their alkaloids. He founded at the Polytechnic School the first Russian factory of pharmaceutical products, which also served for educational purposes.

After 1917 he served as manager of several institutions for the industrialization of the country and for scientific activities. In 1918 Chichibabin organized a chemical department for the War-Industries Committee which established the first sulfuric acid factory at Rastyapin. It is now a large center of chemical industry. Chichibabin was also president of the State Managing Committee of Pharmaceutical Factories and a member of the board of directors for the whole production of chemicals and pharmaceuticals. He was a member of the Scientific Council in the State Department of Health, president and editor-in-chief of the State Pharmacopoea (7th ed.), etc.

During his life he held over 40 different key positions. His activities were so highly appreciated that he was promoted to Grade Five in the Soviet Register of Scientific Workers, among the first 25 scientists from different branches in the whole USSR.

In 1930 he suffered a heavy blow when his only daughter, Natasha, a student of chemistry, met with a fatal accident while working in a factory, when a sulfuric acid container exploded. Chichibabin could not reconcile himself to such a loss and could find no more energy for his daily work. He therefore took a leave of absence, and with his wife, Vera Vladimirovna, he went to Paris.

At Paris he worked in the laboratory of the Hôtel Dieu with Professor Tiffeneau, and later at the Collège de France. At the same time he was Director of the Scientific Department of Kuhlman Co., and technical adviser at Schering and Roosevelt Co. in New York. In 1935 he became an honorary member of the Academy of Science in Torino (Italy). Although his health was weakening, he spent the rest of his life in laboratory work and in compiling his autobiography. He died in Paris at his home on Rue Emile Ducleau, No. 2, on August 15, 1945, leaving 251 scientific works and a total of 346 publications and patents.

On March 11, 1960, in Paris died Vera Vladimirovna, his faithful wife, companion, and collaborator. The couple was buried in the cemetery of Ste. Genevieve de Bois, near Paris.

Contributions to Organic Chemistry

The studies on trivalent carbon. The existence of the trivalent carbon atom was established in 1900 by the American scientist, Gomberg, who prepared triphenylmethyl. Chichibabin proved in his studies that it actually consisted of hexaphenylethane which dissociated in small quantities in solution to triphenylmethyl. This compound is known in the literature as "Chichibabin's hydrocarbon (7)."

In his later work the author developed a hypothesis of the existence of trivalent carbon in compounds of the aromatic and ethylenic series. His theory was accepted by some chemists, such as Debye and Scherrer (in 1917). Observing it in the light of the modern point of view on the entity of chemical bonds, it must be acknowledged that Chichibabin was partly right when he expressed himself on trivalent carbon in compounds with double bonds since they are now considered to be compounds with three equivalent σ bonds, while the fourth is the π bond. Consequently in this theory, as in the case of many previous ones, there is some truth. Obviously at the beginning of the century Chichibabin could not anticipate the existence of two kinds of bonds, σ and π.

Chemistry of pyridine bases. The most important of the studies of Chichibabin were on pyridine compounds. Derivatives of this heterocyclic substance were little investigated before him because of the difficulty of obtaining them. Chichibabin accomplished the synthesis of α- and γ-aminopyridines by the reaction of pyridine with sodium amide. These compounds were easily prepared and were very reactive, in distinction from pyridine itself. This fact made possible the synthesis of a large number of substances of the same series. It was also easy to prepare from the aminopyridines the highly reactive pyridines, which increased the number of pyridine derivatives.

A series of azo dyes were obtained from the same aminopyridines. Some of these found a practical application, as for example 3-phenylazo-2-(2,6-diamino) pyridine, which under the name of "Pyridium" was put on the market as a cure for inflammation of the urinary organs. This was discovered prior to finding the sulfonamides and antibiotics, and for this reason it deserves special attention. The alkylation of aminopyridine revealed the tautomerization among the α and γ pyridine derivatives. This phenomenon is analogous to that of the tautomerism in α and γ hydroxypyridines. The discovery of the tautomeric forms in these compounds made available an explanation of the reaction mechanism for the formation of bicyclic compounds as in the preparation of indolicine from α-methylpyridine and chloroacetaldehyde. The easy method for preparing α-aminopyridine permitted working out in detail the synthesis of the alkaloid coniine in large quantities for industrial purposes.

Some of the pyridine homologues prepared by Chichibabin have the pleasant odor of flowers; others form colloidal solutions and are therefore used as detergents. He synthesized a large number of pyridine derivatives by condensing ammonia with aldehydes and ketones in the presence of catalysts such as Al_2O_3.

Chichibabin ascertained the constitution of beranine which is in fact a derivative of isocumarine. The natural substances among these heterocyclic compounds were unknown until his research. Together with Preobrashensky he initiated the synthesis of pilocarpine which was later completed by his collaborator.

Chichibabin patented a large number of procedures, including the preparation of quinoline from acrolein. He also worked out in detail the method of preparation of the well-known antimalarial, plasmochine. An interesting discovery was that of the phototropy of the pyridine derivatives, such as 2-(2,4-dinitrobenzyl) pyridine which changes its color according to the intensity of light, so that when exposed to strong light it becomes yellow and in weak light, blue. The author believes that this phenomenon is due to the creation of different tautomeric forms.

Naphthenic acids. Extensive studies were carried out on naphthenic acids extracted from Caucasus petroleum. Chichibabin showed, in opposition to the theory of Markovnikov, that naphthenic belonged to

the fatty acid series and contained only a small quantity of cyclopentane and cyclohexane carboxylic acids.

Metalloorganic compounds. Chichibabin was the first to discover the method of preparing aldehydes from the Grignard reagent and orthoformic esters.

Alkylation of the benzene ring. Chichibabin alkylated benzene in the presence of various condensing compounds (H_3PO_4, H_2SO_4, etc.). As a result of this work a series of compounds with very pleasant odors was discovered, and these have had practical application in the production of perfumes.

Miscellaneous. It is also interesting to mention the synthesis of thiodiethylene glycol which was carried out from ethylene oxide and H_2S. The resulting product served as the raw material for a new synthesis of bis(2-chloroethyl)sulfide (Yperite) which gave a purer, and thus more stable, product than was obtained by the other methods known at the time.

Textbook of organic chemistry. Chichibabin considered his "Basic Introduction to Organic Chemistry" to be the masterpiece of his life. The book was written under extraordinarily difficult conditions during the Civil War, when neither food, heating material, nor handbooks were available. He wrote this book, as he used to say, dressed in any old clothing left at home. He was in a hurry to hand it over, sheet by sheet, to the printer in order to receive an additional ration card for food.

The success of his textbook in the USSR was immense. He lived to see the printing in 1932 of 40,000 copies of the fourth edition of his work. The fifth edition, duly completed with the latest theories, was printed in the USSR a few years ago. The book was translated into French and presumably into Ukrainian and Spanish.

There is no doubt that Chichibabin's work was reflected in the world scientific literature and influenced the development of pyridine chemistry. On the basis of this, J. Renault and J. A. Gautier have written their treatise "L'influence des travaux de Tchitchibabin sur le développement de la chimie des dérivés de la pyridine" (*6*).

In 1952 Roger Adams, University of Illinois, informed the widow of Chichibabin that, at the suggestion of Prof. Marszak of Paris, the reactions established by Chichibabin will bear his name.

In the Collège de France there is a prominent display of the substances which he discovered, and according to private information, the room in which he worked will also bear his name.

With the death of Chichibabin a page in the history of chemistry was turned. So passed away a representative of the old classic chemists of Russia. On these men fell the heavy task of creating under difficult circumstances a basis for the scientific development of their country, and in addition they had to take charge of the initiation of the first steps of industrialization of their previously backward country during the first World War and even after the Revolution.

Acknowledgment

I thank Dr. Claire Krymm, the private physician of the late scientist for submitting to me documents on Chichibabin's life and works.

I am also grateful to Dr. Henry M. Leicester, of the College of Physicians and Surgeons of San Francisco, California, for the English modification of my paper as translated from the original Croatian.

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And in addition, Chichibabin's Autobiography, unpublished; and private information from his widow, letters from Dr. C. Krymm, and personal reminiscences.

Nominees for SAMA Award Solicited

Each year an outstanding chemical educator is the recipient of the ACS Award in Chemical Education sponsored by the Scientific Apparatus Makers Association. The Award is $1000 and an appropriately engraved certificate.

A nominee must have made outstanding contributions to chemical education in its broadest sense. Activities recognized by the award include the instructing and training of professional chemists; the dissemination of reliable information about chemistry to the general public, to students of chemistry and of other fields, to members of the profession and to prospective chemists; the integration of science and chemistry into the educational system; establishment of standards and methods of instruction, etc.

Any member of the Society (except a member of the award committee) may submit one nomination for each award.

Eight copies of the nominating materials must be furnished for distribution to the Selection Committee of the ACS Award in Chemical Education; these items must be received in the Office of the ACS Executive Secretary before *April 1, 1962.* Proposals must be accompanied by a biographical sketch of the nominee with specific identification of the work on which the nomination is based, along with a list of publications and patents, and an evaluation and appraisal of the nominee's accomplishments.

The Canvassing Committee (Professor Otto M. Smith, 501 Hillcrest Drive, Stillwater, Okla.; Dr. M. Kent Wilson, Chemistry Dept., Tufts University, Medford, Boston 55, Mass.; and Professor Laurence L. Quill, *Chairman,* Dept. of Chemistry, Michigan State University) are ready to assist anyone who wishes to submit a nomination.

International Atomic Weights — 1961

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Element	Symbol	Atomic number	Atomic weight
Actinium	Ac	89	
Aluminum	Al	13	26.9815
Americium	Am	95	
Antimony	Sb	51	121.75
Argon	Ar	18	39.948
Arsenic	As	33	74.9216
Astatine	At	85	
Barium	Ba	56	137.34
Berkelium	Bk	97	
Beryllium	Be	4	9.0122
Bismuth	Bi	83	208.980
Boron	B	5	10.811[a]
Bromine	Br	35	79.909[b]
Cadmium	Cd	48	112.40
Caldium	Ca	20	40.08
Californium	Cf	98	
Carbon	C	6	12.01115[a]
Cerium	Ce	58	140.12
Cesium	Cs	55	132.905
Chlorine	Cl	17	35.453[b]
Chromium	Cr	24	51.996[b]
Cobalt	Co	27	58.9332
Copper	Cu	29	63.54
Curium	Cm	96	
Dysprosium	Dy	66	162.50
Einsteinium	Es	99	
Erbium	Er	68	167.26
Europium	Eu	63	151.96
Fermium	Fm	100	
Fluorine	F	9	18.9984
Francium	Fr	87	
Gadolinium	Gd	64	157.25
Gallium	Ga	31	69.72
Germanium	Ge	32	72.59
Gold	Au	79	196.967
Hafnium	Hf	72	178.49
Helium	He	2	4.0026
Holmium	Ho	67	164.930
Hydrogen	H	1	1.00797[a]
Indium	In	49	114.82
Iodine	I	53	126.9044
Iridium	Ir	77	192.2
Iron	Fe	26	55.847[b]
Krypton	Kr	36	83.80
Lanthanum	La	57	138.91
Lead	Pb	82	207.19
Lithium	Li	3	6.939
Lutetium	Lu	71	174.97
Magnesium	Mg	12	24.312
Manganese	Mn	25	54.9380
Mendelevium	Md	101	
Mercury	Hg	80	200.59
Molybdenum	Mo	42	95.94
Neodymium	Nd	60	144.24
Neon	Ne	10	20.183
Neptunium	Np	93	
Nickel	Ni	28	58.71
Niobium	Nb	41	92.906
Nitrogen	N	7	14.0067
Nobelium	No	102	
Osmium	Os	76	190.2
Oxygen	O	8	15.9994[a]
Palladium	Pd	46	106.4
Phosphorus	P	15	30.9738
Platinum	Pt	78	195.09
Plutonium	Pu	94	
Polonium	Po	84	
Potassium	K	19	39.102
Praseodymium	Pr	59	140.907
Promethium	Pm	61	
Protactinium	Pa	91	
Radium	Ra	88	
Radon	Rn	86	
Rhenium	Re	75	186.2
Rhodium	Rh	45	102.905
Rubidium	Rb	37	85.47
Ruthenium	Ru	44	101.07
Samarium	Sm	62	150.35
Scandium	Sc	21	44.956
Selenium	Se	34	78.96
Silicon	Si	14	28.086[a]
Silver	Ag	47	107.870[b]
Sodium	Na	11	22.9898
Strontium	Sr	38	87.62
Sulfur	S	16	32.064[a]
Tantalum	Ta	73	180.948
Technetium	Tc	43	
Tellurium	Te	52	127.60
Terbium	Tb	65	158.924
Thallium	Tl	81	204.37
Thorium	Th	90	232.038
Thulium	Tm	69	168.934
Tin	Sn	50	118.69
Titanium	Ti	22	47.90
Tungsten	W	74	183.85
Uranium	U	92	238.03
Vanadium	V	23	50.942
Xenon	Xe	54	131.30
Ytterbium	Yb	70	173.04
Yttrium	Y	39	88.905
Zinc	Zn	30	65.37
Zirconium	Zr	40	91.22

[a] The atomic weight varies because of natural variations in the isotopic composition of the element. The observed ranges are boron, ±0.003; carbon, ±0.00005; hydrogen, ±0.00001; oxygen, ±0.0001; silicon, ±0.001; sulfur, ±0.003.

[b] The atomic weight is believed to have an experimental uncertainty of the following magnitude: bromine, ±0.002; chlorine, ±0.001; chromium, ±0.001; iron, ±0.003; silver, ±0.003. For other elements the last digit given is believed to be reliable to ±0.5.

EDUCATION LIBRARY

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Device to Convert a Hypodermic Syringe into a Multi-Purpose Apparatus

The hypodermic syringe can be used for many purposes other than that of transferring fluids into or out of living organisms. The simple base or holder described in this paper will keep a Luer or other type of hypodermic syringe and its contents in an upright position so that it can be used for the following purposes:

(1) The rapid, accurate weighing and transferring of volatile, viscous, or other types of liquids.

(2) The heating and/or cooling of small amounts of liquids. The entire assembly (Fig. 1A) consisting of the base and the syringe can be placed in a heating or cooling bath until the contents of the syringe reach the desired temperature.

(3) As a substitute pycnometer or specific gravity bottle for the determination of specific gravities.

(4) A series of holders affixed to a rectangular base (Fig. 1B) makes a handy storage rack for partially filled syringes.

(5) As a calibrated pipet or dropping device. Through the use of calibrations on the syringe barrel, amounts as small as 0.01 ml can be accurately delivered.

Essentially the device consists of a flat, circular brass disc approximately two inches in diameter. Affixed to its center is a holder made from that part of the hypodermic needle which fits over the tip of the syringe. The size and shape of the base can be modified to suit any special need.

Placing the tip of the syringe into the holder seals it against loss by evaporation and keeps it in an upright position.

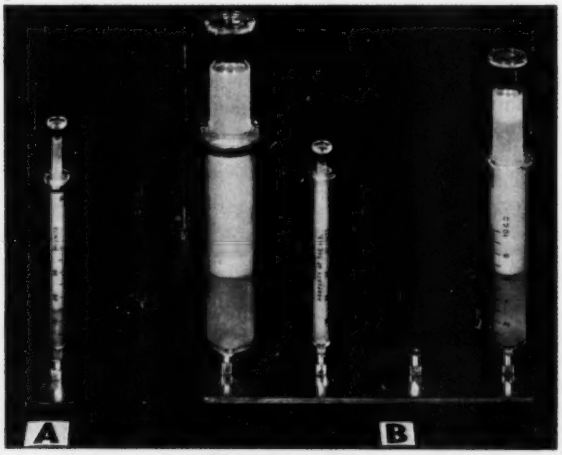

Figure 1. (A) The base and syringe assembly ready for weighing; (B) the base used as a storage rack for partially filled syringes.

In use, the volatile liquid is drawn up into the barrel of the syringe in the usual manner. The needle is removed, the tip of the syringe is then placed in the holder of the utility base, and the entire assembly is placed on the pan of an analytical balance and weighed. The syringe is then removed from the holder, an estimated amount of the liquid needed is ejected into the proper receiver, and the syringe is re-inserted into the same holder and reweighed. The difference between the two weighings is the amount delivered in the receiver.

The use of a large gauge hypodermic needle for filling the syringe keeps the tip clean. A needle should not be used when the liquid is transferred from the syringe into the receiver prior to the second weighing because liquid will be left in the shaft when the needle is removed.

The device can be easily constructed from a circular piece of brass 2 in. in diameter and $^1/_{16}$ in. thick. The shaft of the needle is broken off close to the section that is inserted over the tip of the syringe. The remaining portion of the base is filed smooth and soldered to the metal plate. The soldering should completely encircle the holder so that no air can be admitted. Figure 1B shows a modification of this device being used as a rack for the storage of a number of syringes of varying sizes.

The necessity for the repeated use of the balance can be eliminated when the same amount of the liquid has to be weighed out a number of times. The amount desired is weighed only once and the volume it displaced in the syringe is used for the measuring of subsequent amounts.

When used for specific gravity determinations the syringe is used in the same manner as a pycnometer.

The availability of inexpensive plastic hypodermic syringes brings down the cost of this technique to a matter of pennies, provided the liquids used do not react with the plastic from which the syringe is made. If there is a reaction then the glass type must be used. Glass must also be used in a water bath for the heating of liquids. When used with substances that are difficult to clean the plastic syringe is thrown away. These syringes were originally made for physicians for one time use.

Acknowledgment

Appreciation is expressed to Woodrow Thurston, laboratory mechanic, for fabricating the various holders.

✦ ✦ ✦

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Textbook Errors, 35
Miscellanea No. 4

The Chromyl Chloride Test for Chlorides

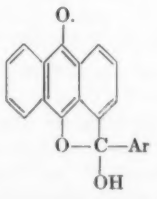
Red droplets of chromyl chloride, CrO_2Cl_2, condense on cooler parts of a test tube in which a mixture of chloride, potassium dichromate, and concentrated sulfuric acid is heated. This is often[1] suggested as a good test for chlorides with the implication that it is specific because the corresponding bromide and iodide do not exist. Ramesh Chandra, lecturer at the D. A. V. College, Jullundur, India, points out that this involves two errors. One is that iodides interfere strongly with this test. As has been shown by Krings (*1*) the presence of iodide in only $^1/_{15}$ the concentration of the chloride completely suppresses the formation of chromyl chloride while free chlorine, which always forms to some extent, now becomes the only product. A possible explanation of this shift as well as other interfering reactions may be found in Feigl's compendium (*2*).

A second error is that chromyl bromide is not a nonexistent compound. Zellner (*3*) presented good evidence that it forms by low temperature reaction of chromyl chloride with excess hydrogen bromide, either alone or in the presence of inert solvents. He was able to isolate a crystalline brown product of over 90% purity and stable[2] at room temperature.

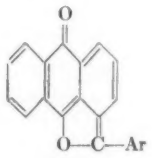
A closely related textbook error which is occasionally encountered is the inclusion of potassium dichromate among the oxidizing agents useful in preparing Cl_2 from HCl or NaCl. Apparently, unless some iodide is also used, CrO_2Cl_2 may be the main product!

"Monovalent Oxygen" Compounds

Compounds having the formula



are cited in advanced tests[1] as examples of stable oxygen free radicals. However, as pointed out by Dr. Yoshio Matsunaga of the University of British Columbia, they do not exist in reality. The compounds were discovered by Schaarschmidt in 1915 (*4*). They were later studied and the above formula assigned to them by R. Scholl on the basis of work (*5*) published between 1921 and 1931. In the following three years a very careful check of the elementary analysis (*6*) led him, however, to revise the structure to the more conventional one:

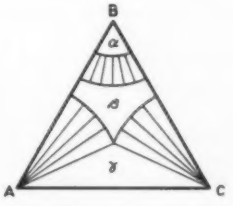


Furthermore, Dr. Matsunaga (*7*) has investigated the phenyl member of this family by electron spin resonance and found no electron spin absorption. This shows the actual absence of any unpaired electrons.

Ternary Diagrams

Professor Scott L. Kittsley of Marquette University calls attention to the fact that three-phase regions of a ternary diagram are frequently[1] labeled as pertaining to two phases only.

In a ternary phase diagram for a given temperature and pressure, such as the one shown in the figure, each point within the triangle corresponds to a given composition of the system in terms of components *A*, *B*, and *C*. Their ratio in the over-all system is given by the distance of the point from the opposite sides of the triangle. Two-phase systems are indicated by tie lines whose ends show the nature of the two phases forming any system whose over-all composition is a point on the tie line. The amounts of the two phases vary, of course, as the position of the point moves along the tie line.



The blank regions may correspond either to one-phase or three-phase systems. One-phase regions, such as α and β, may be recognized generally by the fact that tie lines end on their boundary since they show the composition of these phases in the two-phase region. Furthermore, the boundaries of one-phase regions are generally curved. In contrast, a region such as γ, which is bounded by straight lines substantially parallel to tie lines in any adjacent two-

Suggestions of material suitable for this column and guest columns suitable for publication directly are eagerly solicited. They should be sent with as many details as possible, and particularly with references to modern textbooks, to Karol J. Mysels, Department of Chemistry, University of Southern California, Los Angeles 7, California.

[1] Since the purpose of this column is to prevent the spread and continuation of errors and not the evaluation of individual texts, the source of errors discussed will not be cited. The error must occur in at least two independent standard books to be presented.

[2] Contrary to the summary of *Chem. Abstr.*, **44**, 2879 (1950). The compound was, however, slightly sensitive to light and extremely sensitive to moist air.

phase regions, must be a three-phase region. The composition of the three phases forming all systems whose over-all composition lies within the triangle is given by its three apexes. Thus, in the case of the figure, all systems with over-all compositions lying within γ are formed by pure A, pure C, and β of the particular composition given by the point where the γ and β regions touch.

Discussed Elsewhere

Eutectics. The frequent error of representing eutectic solids as a separate phase on phase diagrams, while in fact they are never compounds but always two-phase mixtures, is pointed out in the extensive article of G. N. Copley on "Eutexia" in the December, 1959, issue of THIS JOURNAL (*8*). He also brings out their structural and metallurgical importance, which underscores the need for the correct treatment of this subject.

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(5) SCHOLL, R., ET AL., *Ber.*, **54**, 2376 (1921); **56**, 918, 1065, 1633 (1923); **60**, 1236, 1685 (1927); **61**, 968 (1928); **64**, 1158 (1931).
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Chemical Information Centers of Latin America

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Summit, New Jersey

Interest in the scientific activities of our colleagues south of the border has been increasing steadily; this interest has been spearheaded by the "Handbook of Latin American Studies,"[2] published yearly since 1936, the source of some information of scientific importance; several review articles have appeared, describing the scientific education in Latin America[3,4]; also, Stevens[5] has given a brief survey of the chemical and related literature of Latin America.[6] This paper, which supplements the information compiled by Stevens in 1957, will be mainly concerned with the centers of chemical and scientific information listed in Table 1. Of these centers, the S.I.D.Q. (Service of Chemical Documentation and Information) is the only one concerned with chemical documentation exclusively; the others are concerned with all the sciences, except for the I.C.A.I.T.I. (Central American Institute for Industrial Research and Technology), which is devoted primarily to services to industry, and the I.P.C.A. (Pan American Institute of Agricultural Sciences), which is devoted to agricultural research.

Center of Scientific and Technical Documentation of Mexico

The C.D.C.T.M. was organized in 1950 by the Mexican Government and UNESCO; after three years of joint operation, UNESCO withdrew and the Center became part of the Secretary of Public Education, although (in the words of its present director, Dr. A. M. Sandoval) "keeping its original philosophy of serving as a link between the Latin American countries and the rest of the world." The Center receives a subsidy from the Mexican Government; the rest of its income is derived through the sale of its services, some of which include literature searches, translations, publication of the *Boletin*, photo-reproduction, and library services.

The *Boletin* is divided into five sections, each published separately every month: (1) Mathematics, Astronomy and Astrophysics, Physics, Geology, and Geodesy; (2) Engineering and Architecture; (3) Chemistry; (4) Medicine; (5) Biology, Agriculture, Husbandry, and Food Industries. Section (3) could be described as a hybrid between *Chemical Abstracts* and *Current Chemical Papers*. Unlike the former it

[1] For reprints: 52 Franklin Place, Summit, N. J.
[2] HANKE, LEWIS, ET AL., editors, "Handbook of Latin American Studies," Harvard University Press, Cambridge, Mass., Vols. 1–22 (1936–1960).
[3] PAUL, J. R., *Yale J. Biol. and Med.*, **31**, 284–93 (1959).
[4] JAMES, A. E., *Am. J. Pharm. Educ.*, **22**, 51–61 (1958).
[5] STEVENS, L. J., "J. CHEM. EDUC.," **34**, 90–4 (1957).
[6] The name Latin America is used here instead of South America in deference to the custom in that region, since this survey covers countries from Mexico to Argentina. The term South America is reserved for the territory encompassed by Colombia to Argentina.

does not give abstracts of all the papers, and it is more comprehensive than the latter because it gives a summary in English of the papers published in Latin America; those originating from this region are identified by a black dot. The chemical section follows closely the pattern of *CA* in its organization of the subject matter. So far there is no index, but the first one is due to appear sometime this year.

Table 1

Name	Abbreviation	Country
Centro de Documentacion Cientifica y Tecnica de Mexico	C.D.C.T.M.	Mexico
Servicio de Informacion y Documentacion Quimicos	S.I.D.Q.	Peru
Instituto Brasileiro de Bibliografia e Documentacao	I.B.B.D.	Brazil
Instituto Centro Americano de Investigacion y Tecnologia Industrial	I.C.A.I.T.I.	Guatemala
Comision Nacional de Documentacion	C.N.D.	Argentina
Instituto Pan Americano de Ciencias Agricolas	I.P.C.A.	Costa Rica
UNESCO Centro de Cooperacion Cientifica para la America Latina	C.C.C.L.A.	Uruguay

Service of Chemical Documentation and Information

This service was established by the Board of Directors of the Chemists' Association of the National University of San Marcos in 1956. This is the only one of the institutions here described which is strictly private. Its aims are to catalogue all chemical information from Peruvian and foreign sources and to make it available on request to students, researchers, chemical industries, and other interested parties. All its services are free. It also serves as a link between the national and foreign chemists. To date it does not have any periodical publications; occasionally, however, it publishes papers pertaining to topics of special interest. Other tasks are to collect materials for the history of chemistry in Peru, to organize chemical libraries in the universities, and to prepare courses on Chemical Documentation. It also maintains a collection on Peruvian chemical history in the Smith Memorial Collection of the University of Pennsylvania.

Brazilian Institute of Bibliography and Documentation

The I.B.B.D. was established as a department of the Brazilian Research Council in 1954 with the cooperation of UNESCO, the Getulio Vargas Foundation, and the Brazilian Institute of Education, Science, and Culture. Its purposes are to serve as a center of scientific information, to organize a Brazilian scientific bibliography, to prepare translations and literature searches, to provide microfilms, and to maintain a cooperative cataloguing service. The Institute has published, so far, Brazilian Bibliographies on Chemistry, Mathematics, Physics, Documentation, Botany, Zoology, and Medicine.

Central American Institute for Industrial Research and Technology

This institute is an industrial consulting organization rather than a scientific information center. It is active in the fields of industrial research and development, utilization of mineral and agricultural materials, market surveys, and the like. It also undertakes laboratory and quality control tests. The Institute was organized with the financial assistance of the Technical Assistance Administration and the Special Fund of the United Nations. Its personnel are truly international: the director is the Austrian Chemist, Dr. Otto J. Stern, and the rest of the staff includes scientists from France, Germany, United Kingdom, and India, as well as Costa Rica, El Salvador, Honduras, Nicaragua, Panama, and Guatemala, which are the six countries this organization serves. It furnishes technological information in fields such as fats and oils, food products, fertilizers, insecticides, textiles, and the heavy chemical industry.

National Commission of Documentation

A National Commission of Documentation was organized in 1960 by the School of Sciences of the University of Buenos Aires, under the direction of Dr. Eduardo L. Ortiz.

Pan American Institute of Agricultural Sciences

The I.P.C.A. is one of the agencies of the Organization of American States, organized in 1942, in order to stimulate the development of the agricultural sciences in the Americas through teaching and research. While this is the main aim of the Institute, it also furnishes some library services such as microfilms, photostats, and short bibliographic surveys. Of chemical interest are their researches on nutrition, insecticides, fertilizers, and plant growth regulators.

Center of Scientific Cooperation for Latin America

The UNESCO C.C.C.A.L. was established in Montevideo in 1949, with the objective of bringing to Latin American scientists scientific information from other parts of the world and also of making known the Latin American scientific achievements within and outside the region. The first objective is accomplished by a Reference Service which has a collection of periodicals (including 800 from Latin America), a reference library, and a card index of Latin American scientific institutions and scientists. The second objective is realized by publications which include 26 volumes of "Scientific Institutions and Scientists of Latin America," a directory of government and private scientific institutions which also gives brief biographies of individual scientists; "Lists of Scientific Articles Published in Latin America," and the series "Latin American Contributions to Scientific Progress" (Chemistry, 1951; Mathematics, 1951; Parasitology, 1953). Bi-monthly a *Boletin* is published giving information of scientific meetings and publications.

Acknowledgment

The author wishes to acknowledge the cooperation of Mrs. Lelia Galvao C. da Cunhua and Mrs. Eva V. Chesneau; Doctors A. M. Sandoval, A. Establier, A. Alcalde-Mongrut, and Otto Stern; and of Mr. H. Calvert Anderson.

B. R. Siebring
The University of Wisconsin
Milwaukee

Institutional Influences in the Graduate Training of Eminent Chemists

Defining the term "eminent chemist" is an extremely difficult and hazardous task. Certainly no individual could arrive at a definition satisfactory to everyone. However, various organizations and publications have methods of recognizing outstanding accomplishments in chemistry. By making use of these recognitions of achievement it is possible to arrive at a list of eminent chemists who have been selected by authorities in the field. This, of course does not eliminate entirely the judgment of the writer, since it was necessary to select the awards and recognitions.

For the purposes of this study, chemists in one or more of the following categories have been classified as eminent chemists:

1. Nobel prize winners.
2. Members of the chemistry division of the National Academy of Science (1948–1960).
3. Persons listed as Distinguished Chemists in the first edition of "Leaders in American Science."
4. Winners of a recognized American award for accomplishments in chemistry.

The reader will be familiar with the citations of the first two categories. The membership of the National Academy of Science has been reported by division in the *Proceedings of the National Academy of Science* since 1948.

The third category can best be described by quoting from the preface of Volume I of "Leaders in American Science." In speaking of their Poll of Distinguished Scientists, the authors state:

> The purpose of our compilation is to list the opinions of the scientists themselves as to the most distinguished men and women in their own specialized fields . . . Again, this list of distinguished scientists is not of our own choosing; it is based on the choices of thousands of reputable scientists who themselves did the choosing.

The list of recognized American awards in chemistry was compiled from lists in "Handbook of Scientific and Technical Awards in United States and Canada 1900–1952"[2] and "The Blue Book of Awards."[3] Those awards which were primarily for the purpose of education or training were not included. Of course, many names appeared several times, some as many as seven or eight times. For example, Glenn T. Seaborg was listed in all four categories and five times in the fourth category.

Certainly it can be argued that there are eminent chemists who have not acquired any of the distinctions in the above categories. However, it seems reasonably safe to conclude that chemists in the above categories have made significant contributions to chemistry and are correctly classified as "eminent chemists." These categories are sufficiently diverse so that chemists from almost any field could qualify, although the ease of qualification is probably not the same in all areas of chemistry. Nevertheless, the writer felt that the four categories were sufficiently widespread so that they could be used as a means of comparing institutions as doctorate sources of eminent chemists.

Information as to where these individuals received their doctorates was obtained from various biographical sources such as "American Men of Science," "Chemical Who's Who," and *Chemical and Engineering News*.

Four hundred fifty-six eminent chemists received doctorates from American universities. The distribution for the leading doctoral sources of eminent chemists is presented in Table 1. One out of 12 received his Ph.D. degree from Harvard. Approximately 117 got their doctoral degrees from one of the top 10 institutions.

Table 1. Doctorate Origins of 456 Eminent Chemists

Institution	Number
Harvard	37
Chicago	34
Columbia	32
Illinois	32
California	31
Wisconsin	24
Yale	24
Princeton	22
Cal. Tech.	21
Cornell	19
Johns Hopkins	18
Michigan	16
Ohio State	15
Mass. Inst. Tech.	14
Minnesota	14
Pennsylvania	11

If the chemistry members of the National Academy of Science are considered as a group by themselves, the doctorate training is concentrated even more. Of the 117 in this category, 70 received their graduate training in the top six universities listed in Table 2.

The writer has published a paper evaluating undergraduate chemistry departments by determining the proportion of baccalaureate chemistry graduates who later earned a Ph.D. in chemistry.[4] In the present

[1] Cook, Robert C., "Leaders in American Science," 1st ed., Who's Who in American Education, Inc., Nashville, Tennessee 1955.

[2] Firsh, Margaret A., "Handbook of Scientific and Technical Awards in United States and Canada, 1900–1952," Special Libraries Association, New York, 1956.

[3] Brook, Herbert, "The Blue Book of Awards," Marquis Who's Who, Chicago, 1956.

[4] Siebring, B. R., "Institutional Influences in the Undergraduate Training of Ph.D. Chemists," J. Chem. Educ., 31, 195–200 (1954).

Table 2. Doctorate Origins of Chemistry Members of the National Academy of Sciences (1948–1960)

Institution	Number
Harvard	18
California	14
Columbia	12
Cal. Tech.	10
Illinois	8
Wisconsin	8
Mass. Inst. Tech.	7
Yale	5
Chicago	4
Pennsylvania	4
Princeton	4

article the writer has attempted to evaluate graduate departments in a similar fashion by determining the proportion of doctorate graduates who later received the distinction which classified them as eminent chemists as defined above. The data for the leading institutions in the 1930–39 and 1940–49 decades is listed in Table 3. This list includes only those institutions which had three or more doctorate graduates who later qualified as eminent chemists. The number of chemistry doctorates granted by each institution for the 1930's was obtained from *A Guide to Colleges, Universities and Professional Schools in United States.*[5] The data for the 1940's was obtained from Progress Reports published by the ACS Committee on Professional Training in the *Chemical and Engineering News.*

Table 3. Institutions Ranked by Proportion of Chemistry Doctorates, 1930–1949, Who Later Received Recognitions Which Classified Them as Eminent Chemists

Institution	Eminent chemists (1)	Total no. of doctorates (2)	$\frac{(1)}{(2)} \times 100$
Cal. Tech.	12	137	8.8
Princeton	13	198	6.6
Harvard	16	265	6.0
California	17	311	5.5
Brown	3	89	3.4
Chicago	12	383	3.1
Minnesota	8	289	2.8
Wisconsin	14	519	2.7
Yale	6	253	2.4
Pennsylvania	3	132	2.3
Columbia	10	453	2.2
Michigan	6	267	2.2
Iowa State	5	235	2.1

By consulting the ACS "Directory of Graduate Research for 1959,"[6] the writer determined the employment of eminent chemists by institution for that year. The leading institutions in terms of number of eminent chemists employed are listed in Table 4.

Table 4. Institutional Employment of Eminent Chemists, 1959

Institution	Number
Harvard	12
California (Berkeley)	10
Cal. Tech.	9
Columbia	9
Illinois	9
Iowa State	8
Mass. Inst. Tech.	8
Wisconsin	7
Chicago	5
Northwestern	5
Penn State	5

The distribution of employment for the chemistry members of the National Academy of Science is presented in Table 5.

Table 5. Institutional Employment of Chemistry Members of National Academy of Science, 1959

Institution	Number
Harvard	8
California (Berkeley)	6
Cal. Tech.	5
Illinois	5
Wisconsin	5
Mass. Inst. Tech.	4
Columbia	4
Minnesota	3
Cornell	3

Table 6 lists the leading institutions by percentage of staff members classified as eminent chemists. The total number of staff members was determined by counting the professors listed in the ACS Research Directory for 1959.[6] Similar data for the National Academy of Science chemistry members (exclusively) is presented in Table 7.

Table 6. Institutions Ranked by the Proportion of Chemistry Staff Classified as Eminent Chemists (1959)

Institution	Eminent chemists (1)	Total (2)	$\frac{(1)}{(2)} \times 100$
Columbia	9	21	42.9
Harvard	12	28	42.9
Cal. Tech.	9	29	31.0
Virginia	4	13	30.8
California (Berkeley)	10	37	27.0
Mass. Inst. Tech.	8	33	24.2
Northwestern	5	21	23.8
Wisconsin	7	30	23.3
Princeton	4	21	19.0
Illinois	9	48	18.8
Chicago	5	27	18.5

Table 7. Institutions Ranked by the Proportion of Chemistry Staff who are Members of the Chemistry Division of the National Academy of Science

Institutions	Academy members (1)	Staff total (2)	$\frac{(1)}{(2)} \times 100$
Harvard	8	28	28.6
Columbia	4	21	19.0
Cal. Tech.	5	29	17.2
Wisconsin	5	30	16.6
California (Berkeley)	6	37	16.2
Rochester	2	13	15.4
Cornell	3	22	13.6
Mass. Inst. Tech.	4	33	12.1
Illinois	5	48	10.4
Princeton	2	21	9.5

The *Chicago Tribune* in its articles on the nation's top ten universities selected California (Berkeley), Chicago, Yale, Harvard, Wisconsin, and Cornell as the universities with outstanding departments in chemistry.[7] These choices were based on the eminence of the faculty in these departments. Cal. Tech. and

[5] Good, Carter V., "A Guide to Colleges, Universities, and Professional Schools in the United States," American Council on Education, Washington 6, D.C., 1945.

[6] American Chemical Society, "Directory of Graduate Research," 1959.

[7] Manly, Chesly, Chicago Sunday *Tribune*, April 21, 1957.

MIT were not considered as universities by the Chicago writers. The reader will note that four of these universities (California, Chicago, Harvard, and Wisconsin) rank high in nearly every ranking of this study. There is not such complete agreement concerning Yale and Cornell. Columbia ranked high in nearly all situations but was not listed by the Chicago writers as an institution with a top chemistry faculty.

Irwin Scher, Edward Walsh,
and **Barney Kadis**[1]
State University of New York
College of Education, Albany

A Biochemistry Laboratory Experiment

We have found that students in an advanced course in biochemistry enjoy the challenge of performing an experiment from the current literature or devising a problem of their own. Such an experiment is described here. Discussion of the origins of life on earth stimulated the interest of one student to perform a duplication of the Miller experiment.[2] This unique experiment was designed to simulate prebiological conditions for amino acid synthesis, using an electrical discharge in a reducing atmosphere.

Construction of the apparatus (Fig. 1) posed no serious problems. Instead of using the coil suggested by Miller for the source of electrical discharge, an induction coil (Cenco 7985) was substituted. Since yields reported by Miller were not enhanced by the presence of hydrogen gas, safety considerations suggested that hydrogen be omitted from the reaction mixture. Instead of pure methane, ordinary burner gas (methane, 95.1%; ethane, 3.8%; other, 1.1%) was substituted. Otherwise the original experimental conditions were followed. The reaction was performed in a fume hood surrounded by safety shields. After seven days the reaction was stopped and the water removed. The apparatus was washed with additional water, and the combined washings filtered. The solid material appeared to be inorganic (ignition test). The brown aqueous phase was clarified with decolorizing charcoal. Some amino acids may have been lost due to absorption on the charcoal, but because of the simplicity of the procedure, this objection was disregarded.

One-dimensional chromatograms were prepared using a butanol-acetic acid-water system. Spots corresponding to glycine, alanine, and possibly aspartic acid were detected with ninhydrin spray. Two-dimensional chromatograms following the procedure of Cowgil and Pardee[3] afforded additional evidence for the presence of alanine and glycine. Aspartic acid still remained elusive.

A simple electrophoresis apparatus[4] was constructed from a power supply, carbon electrodes, glass plates, and beakers. A solvent system of 70% acetic acid was used. At a voltage of approximately 500 dc volts, the separation was allowed to proceed for four hours, with the definite presence of alanine and glycine demonstrated. Again, an area corresponding to aspartic acid was found.

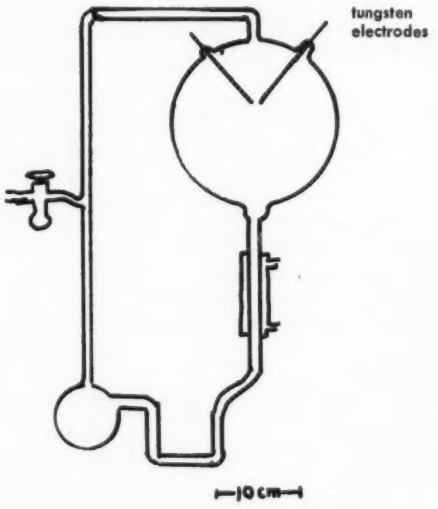


Figure 1. Reaction vessel.

Conclusive evidence for the synthesis of alanine and glycine was obtained by means of their 2,4-dinitrobenzene derivatives. The compounds were prepared using 1-fluoro-2,4-dinitrobenzene according to the directions of Cowgil and Pardee. Separation by desending paper chromatography led to R_f values which corresponded to known glycine and alanine derivatives which were run simultaneously. A spot corresponding to aspartic acid was present, but the R_f value was somewhat ambiguous. Infra-red spectrum (KBr pellet) of the crude residue also confirmed the presence of amino acids.

The experiment proved valuable as a means of combining the teaching of basic techniques of biochemistry with stimulating interest in a theoretical problem—the synthesis of amino acids.

The valuable discussions with Dr. Charles Ise are gratefully acknowledged.

[1] Present address: Dept. of Biochemistry, U. of Nebraska College of Medicine, Omaha 5, Neb.

[2] MILLER, S. L., *J. Am. Chem. Soc.*, **77**, 2351 (1955).

[3] COWGIL, R. W., AND PARDEE, A. B., "Experiments in Biochemical Research Techniques," John Wiley and Sons, Inc., New York, 1957, p. 46.

[4] LEDERER, M., "An Introduction to Paper Electrophoresis and Related Methods," Elsevier Publishing Co., New York, 1955, p. 41.

John T. Stock
University of Connecticut
Storrs

A Titration Controller for Demonstrating the Behavior of Acid-Base Indicators

To demonstrate the behavior of typical acid-base indicators, a controller based on general-purpose transistors such as Raytheon CK 722 or General Electric 2N107 was developed. The device permits the automatic back-and-forth titration with acid and alkali of a solution containing an appropriate indicator.

The controller has been operated in conjunction with a Beckman H2 or a Leeds and Northrup 7664 *p*H meter equipped with the usual glass-calomel electrode pair. In the case of the Beckman meter, input was taken across the binding posts of the galvanometer movement. Although the Leeds and Northrup meter has an outlet for a 20-millivolt recorder, the available signal is too small for the present purpose. A much larger and very satisfactory signal is however obtainable from the "automatic temperature compensator" outlet, which becomes operative when the compensator switch is moved from "manual" to "automatic." If this method is used, a resistance R_6 of about 800 ohms should be connected across the controller input. Since this resistance governs the correction of the *p*H readings, the latter will be exact at one temperature only. In the back-and-forth titration over a range of about 2 *p*H units, this small possible *p*H error is immaterial.

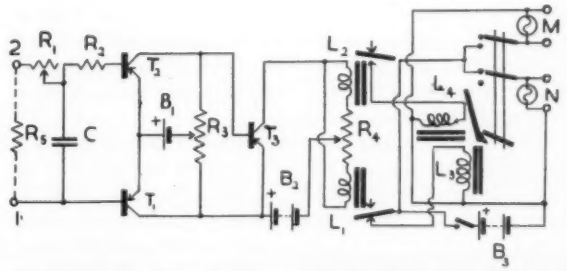
Input from the *p*H meter is connected so that binding post 1 becomes increasingly negative as alkali enters the solution being titrated. Since the sensitivity control R_1 needs adjustment only when a different *p*H meter is used, this resistor is a screwdriver-operated one. Zero control R_3 backs off the input signal, so that the controller responds only to changes in this signal that occur during titration. The amplified signal is divided unequally between sensitive relays L_1 and L_2 by means of radio potentiometer R_4. This enables L_1 to be set to pull in ahead of L_2 and to drop out after it, thus providing for the successive energizing of the unlatching and latching coils L_4 and L_3 of the latching relay.

Flashlamp cells are used for batteries B_1 and B_2. The current drawn from these is only a milliampere or so and expiration of shelf life is the usual cause of replacement. Since a current of 1–2 amperes at 6 volts is required to operate the latching relay and the reagent control valves, a storage battery or line-operated dc supply should be used for B_3.

Any of the various forms of electrically-operated reagent delivery systems (*1*) may be used with this apparatus. Typical home-made forms incorporate a commercial magnetic stirrer-bar (*1*) or a strip-iron lever (*2*) as moving elements.

Suppose that the electrodes are inserted in a slightly-acid solution and that R_3 is set so that the amplified signal is insufficient to cause either L_1 or L_2 to pull in. Current from B_3 then flows through the contacts of L_1, energizes coil L_3 of the latching relay, and causes the double-throw contacts associated with this coil to pass current from B_3 to binding posts M. Since the alkali control valve is connected to these binding posts, "forward" titration commences. At the same time, a pilot lamp connected across M illuminates a small window marked "ADDING ALKALI."

As alkali flows, the emf applied to the controller increases until relay L_1 pulls in. Although power to coil L_3 is now cut off, the contacts of the latching relay do not move and titration continues. Eventually, the emf becomes high enough to cause the pull in of relay L_2. Coil L_4 is then energized, the latch retracts and the double-pole contacts spring back to the normal position shown. This transfers the power supply to binding posts N, which are connected to the acid control valve and to a second lamp that signals "ADDING ACID." "Forward" titration is thus arrested as "back" titration commences. As the input emf falls, relay L_2 drops out, but does not influence the position of the contacts of the latching relay. Continued flow of acid finally causes relay L_1 to drop out, with consequent energization of coil L_3. Addition of acid is then arrested and that of alkali is restarted. Once the controls have been appropriately set, alternate "forward" and "back" titrations continue automatically.



Circuit diagram for titration controller. B_1, 1.5 volts; B_2, 15 volts; B_3, 6 volts; C, 100 μF electrolytic capacitor; R_1, 100K; R_2, 10K; R_3, 10K (preferably; but not essentially, 10-turn); R_4, 10K; R_5; see text; L_1, L_2, Kurman type 31002/42Y or similar sensitive relay; L_3, L_4, coils of Potter and Brumfield KB17DG or similar latching relay; T_1, T_2, T_3, General Electric 2N107 or similar transistor.

In a typical demonstration, a 2-liter beaker is filled to about three quarters with 0.2 *M* sodium chloride solution. Sufficient bromothymol blue solution is added to give a pronounced coloration, the electrodes are inserted, and the mixture is stirred magnetically. The controller is then set to titrate back and forth between the approximate *p*H limits 5.5 and 8.0. Titrants, 0.1 *M* hydrochloric acid and 0.1 *M* sodium hydroxide (concentrations approximate), are run in at about 30 drops per minute.

Because of its spectacular yellow-green-blue color change as the *p*H changes, bromothymol blue is very satisfactory. Since its *p*H 6.0 to 7.6 working range spans neutrality, only about 20 drops of acid or alkali are required to produce a visibly complete color change. Methyl red, of working range *p*H 4.2 to 6.3, requires nearly double the amount of titrant.

When a *p*H meter is not available, the glass electrode may be replaced by an antimony electrode. Such an electrode may be cast in 10-mm test tube, mounted on the end of a length of glass tubing (*3*), and connected directly to binding post 1. A saturated calomel or silver-silver chloride reference electrode is attached to the other binding post. The antimony electrode is precise only under carefully controlled conditions (*4*, *5*) and responds sluggishly to *p*H changes. The emf change in continuous back-and-forth titration is therefore considerably smaller than in comparable point-by-point titration. Nevertheless, reasonable results are obtainable if the rate of titrant addition is reduced. If the exhibit is merely an eye-catching one, titration over the wider range of about 4 *p*H units using "universal indicator" (*6*) is more satisfactory.

Although developed for acid-base work, the controller can of course be used for other back-and-forth titrations. Particularly when the emf change is small, the sensitive relays should pull in at a current not much greater than the drop out value and should have characteristics as nearly alike as possible.

This apparatus was developed with the partial support of the National Science Foundation's program for the design of science teaching equipment.

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(2) Stock, J. T., and Fill, M. A., *Laboratory Practice*, **10**, 302 (1961).
(3) Stock, J. T., *School Sci. Rev.*, **32**, 287 (1951).
(4) Stock, J. T., Purdy, W. C., and Garcia, L. M., *Chem. Revs.*, **58**, 611 (1958).
(5) Garcia, L. M., Purdy, W. C., and Stock, J. T., *Chemist-Analyst*, **47**, 36 (1958).
(6) Milton, R. F., and Waters, W. A., "Methods of Quantitative Micro-Analysis," 2nd ed., Edward Arnold, Ltd., London, **1955**, p. 411.

A Convenient Safety Shield for Small-Scale Apparatus

Stanley H. Langer[1]
and **Peter Pantages**
U. S. Bureau of Mines, Pittsburgh, Pa.

A light, portable, and convenient shield for small-scale chemical operations can be constructed from a modified, commercially available face mask attached to a swivel mechanism. The swivel mechanism is attached to a rod which is readily connected to a rack or ringstand by a clamp holder.

The shield is locked in place or unlocked with a spring-loaded pin. It may be locked in any one of three positions (upright, horizontally, or down). This allows the shield to be used in various ways and also makes possible moving it out of the way while making apparatus changes. Engineering drawings for the component parts of this shield are available upon request from the Bureau of Mines, 4800 Forbes Avenue, Pittsburgh 13, Pa. Components are fabricated from steel and aluminum.

[1] Present address: American Cyanamid Co., Stamford, Conn.

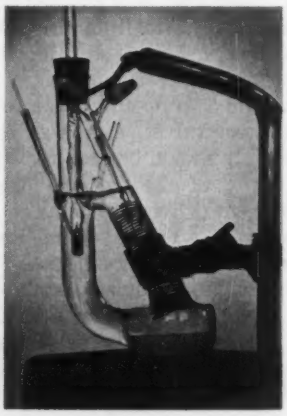
Melting Point Apparatus

Carlton S. Graves
University of Minnesota, St. Paul

The unique feature of the melting point device shown in the accompanying photograph is the centrifugal pump which circulates oil through the system. The Teflon-covered magnetic stirring bar rotates in the small cylindrical chamber. Centrifugal force impels the oil out the side port and up past the thermometer bulb (against which the melting point capillaries rest) and into the heated-oil expansion chamber. From there the oil returns past the Nichrome wire heating element down to the pump chamber.

Dow Corning silicone fluid 550 is used because it does not discolor or smoke at high temperatures. The current to the heating coil is controlled by a variable transformer. A Teflon stopper holds the thermometer in place.

In practice the apparatus is almost vibration free, the oil heats up and cools off quickly, a constant or slowly changing temperature is easily maintained, and the rapid circulation of oil assures quite uniform temperatures in the area of the thermometer bulb.



. . . from the TREASURER

WILLIAM G. KESSEL

Supporting the vigorous program of the Division of Chemical Education is a sound financial structure. Member responsibility to this structure starts with faithful payment of annual dues of $1.50 or for associate membership, $2.00, payable to the Treasurer, January 1 of each year. It goes beyond this to include an approving knowledge of the fiscal policy of the Division. This Treasurer's Report provides each member an opportunity to review this policy and, when confronted with a question, an occasion to write the Treasurer for clarification.

DIVISION ACCOUNT

Receipts:		
Balance, September 1, 1960	$ 1,794.03	
Dues	3,100.25	
Total receipts		$ 4,894.28
Expenditures:		
Printing	878.24	
Postage	714.84	
Office Supplies	190.48	
Office Services	311.00	
Committee operations	391.98	
Journal subscription (foreign)	15.00	
Bank charges	6.40	
Total expenses		2,507.94
Balance September 1, 1961		$ 2,386.34
Assets:		
Balance, according to checkbook, September 1, 1961, First National Bank, Terre Haute, Indiana		$ 2,386.34
Four U. S. Savings Bonds, Series J, Nos. C160832, -3, -4, -5 (maturity value $100 each, May 1, 1967)		400.00
Total assets		$ 2,786.34

JOURNAL ACCOUNT

Receipts:		
Balance, September 1, 1960	$ 9,960.81	
Interest on U. S. bonds	510.80	
Bank dividend and interest	231.92	
Chemical Education Publishing Co.	11,157.35	
Total receipts		$21,860.88
Expenditures:		
Purchase five shares ACS Cash and Investment Pool	$ 5,000.00	
Chemical Education Publishing Co.		
Journal extra pages 1959	531.53	
Journal extra pages 1960	1,389.96	
Total expenditures		6,921.49
Balance September 1, 1961		$14,939.39
Assets:		
Account No. 32596 Fort Harrison Savings Association, Terre Haute, Indiana		$14,939.39
Inventory of securities on deposit with R. V. Mellefont, Treasurer of the ACS:		
Four U. S. Savings Bonds Series G, $1000 each, Nos. M7279936G, M7280064G, M7280066G, and M7280067G, maturing 12-1-61		4,000.00
Three U. S. Savings Bonds, Series G, $1000 each, Nos. M7437661G, M7438009G, and M7438010G, maturing 4-1-62		3,000.00
Eight U. S. Savings Bonds, Series K, $1000 each, Nos. M399072K to M399079K, maturing 7-1-67		8,000.00
Four U. S. Treasury Notes, $1000 each. Nos. 45920 to 45923. Maturing 5-15-64		4,000.00
Five shares, ACS Cash and Investment Pool, purchased 1-2-59		5,344.10
Seven shares, ACS Cash and Investment Pool, purchased 1-2-60		7,685.46
Cash credit in ACS Cash and Inment Pool as of 7-31-60		362.00
Five shares, ACS Cash and Investment Pool, purchased 1-1-61		5,000.00
Total assets		$52,330.95

This does not include credit on shares for 1961.

Report of the New England Association of Chemistry Teachers

Official Business

Meetings

Under the direction of President Carl P. Swinnerton of the Pomfret School, Pomfret, Conn., the Association held five one-day meetings and a six-day summer conference during the year 1960–61. The meetings, as arranged by the Division Chairmen, were as follows:

307th Meeting: Northern Division, October 8, 1960 at the Winnacunnet High School, Hampton, N. H., with Christie J. Drago, Exeter High School, Exeter, N. H., as Chairman.

308th Meeting: Central Division, December 3, 1960, at the Wentworth Institute, Boston, Mass., with Harry G. Stubbs of Milton Academy, Milton, Mass., as Chairman.

309th Meeting: Southern Division, February 11, 1961, at the Moses Brown School, Providence, R. I., with Erich A. O'D. Taylor of the Portsmouth Priory School, Portsmouth, R. I., as Chairman.

310th Meeting: Western Division, April 8, 1961, at St. Joseph College, West Hartford, Conn., with Angela M. Trovato, of Branford High School, Branford, Conn., as Chairman.

311th Meeting: (Annual Meeting): Central Division, May 12, 1961 at Assumption College, Worcester, Mass., with Harry G. Stubbs again as Chairman.

The *Twenty-third Summer Conference* was held August 21–26, 1961, at the University of Vermont, Burlington, with Donald C. Gregg of the University as *Host.* The Co-Chairmen were *Program:* Jay A. Young, King's College, Wilkes-Barre, Pa.; *Arrangements:* Richard L. Saville, Staples High School, Westport, Conn. Pearle R. Putnam, MacDuffie School, Springfield, Mass., was Registrar-Treasurer; and Constance L. Brown, University of Vermont, was Conference Secretary.

The enrollment was 263, the second largest conference of the entire series. Registrants attended from 19 states, the District of Columbia, and two Canadian Provinces. Six registrants present have attended all 23 of the summer conferences:

Mr. and Mrs. Grover C. Greenwood (retired), Bridgeport, Conn.
Mr. Alfred R. Lincoln, Hamden College of Pharmacy, Willimansett, Mass.
Miss Evelyn L. Murdock, Stonington High School, Stonington, Conn.
Miss Elsie S. Scott, Northfield School for Girls, Northfield, Mass.
Mr. John R. Suydam (retired), Boston, Mass.

Appointments and Elections

The Officers elected by the membership for the fiscal year 1961–62 were published in the *Report:* THIS JOURNAL, **38,** 378 (1961). Robert D. Eddy was elected an Honorary Member. Harold A. Iddles, University of New Hampshire, Durham, was made an Emeritus Member.

Appointments for the year include:

Editor of the Report: Robert D. Eddy, Tufts University, Medford, Mass.
Editor of the News-Letter: Howard I. Wagner, State Director of Science Education, Concord, N. H.
Assistant Editor of the News-Letter: Angela M. Trovato, Branford High School, Branford, Conn.
Publicity Agent: Everett F. Learnard, Norwood High School, Norwood, Mass.
Auditor: Laurence S. Foster, Watertown Arsenal, Watertown, Mass.

A *Committee on Revision of the Constitution and By-Laws* was formed including: Chairman, Gordon G. Evans, Tufts University, Medford, Mass.; Ralph E. Keirstead, Connecticut Dept. of Education, Hartford; and Norman G. Mills, Wachusett High School, Holden, Mass.

Marco H. Scheer, Earlham College, Richmond, Ind. (on leave from Nashua High School, Nashua, N. H.) was appointed to the *Necrology Committee.*

Activities

During the year a new set of By-Laws relating to Emeritus Membership were adopted. The Budget Committee drew up a new form of the Budget, hoping in this way to present a better picture of the varied activities of the Association, activities which involve the expenditure of over $4500. It was adopted by the Executive Committee. The 22nd (1960) Summer Conference closed its books with a net profit of $3.67. The year's activities, as a whole, resulted in a profit for the Association of $206.00. At the present time the Endowment Fund has a value of $3085.44.

Membership continues to grow, with 104 new applications acted upon favorably by the Membership Committee. More withdrawals than usual cut down the net gain to 41. In all, 2564 members have been admitted to the Association since its founding in 1898.

A large and valuable collection of *Reports* and other materials was received by the Association from the estate of the late S. Walter Hoyt. Materials of great historic interest have thus been

Twenty-third Summer Conference

added to the archives. The *Reports*, by direction of the Executive Committee, are being used to add to collections of the *Report* in various colleges and universities. Complete sets of this publication are extremely rare.

Looking Ahead

The 24th summer conference will be held August 20–25, 1962, at the University of Massachusetts, Amherst.

The New President

Dr. Leallyn B. Clapp, Professor of Chemistry at Brown, took office on July 1, 1961 as 35th President. A native of Paris, Ill., Dr. Clapp graduated from the Eastern Illinois State Teachers College in 1935 with the degree of Bachelor of Education. He went on to the University of Illinois, where he took the M.S. and Ph.D. degrees in chemistry.

He began his career at Brown as an instructor in organic chemistry. He was made an assistant professor in 1945, an associate professor in 1949 and a full professor in 1956. He was awarded the degree of Master of Arts, *ad eundem*, in 1956, and in the same year received the honorary degree of Doctor of Pedagogy from Eastern Illinois State Teachers College.

Dr. Clapp began his active interest in NEACT almost immediately upon joining the Brown faculty. In 1943 he accepted an appointment as Editor of the *Report*. In 1946 and 1947 he was Chairman of the Southern Division. He has been active in numerous committees, including several summer conference committees. During the period 1951–56 he served as Secretary of the Association, and has been Vice-President since 1959.

Outside of the Association he has been involved in many activities related to chemical education: a member of the Chemistry Committee of the CEEB and active in the development of Advance Placement courses in chemistry, and he has just completed a term of office as Chairman of the ACS Division of Chemical Education. Currently, his favorite activity is that of author and director of the CBA Project. Dr. Clapp is the author of a chapter on Organic Molecular Compounds in "Chemistry of the Coordination Compounds," by John C. Bailar, Jr. He has published his own book "The Chemistry of the Covalent Bond," and has contributed numerous scientific papers on both inorganic and organic chemistry.

In 1940, he was married to the former Florence Cottingham of Charleston, Ill. They live with their two children close to the Brown campus in Providence.

ROBERT D. EDDY
Editor, Report of the NEACT

TUFTS UNIVERSITY
MEDFORD, MASSACHUSETTS

ChemEd

LETTERS

To the Editor:

I read with interest Karl L. Lockwood's article, "Redox Revisited," in the June issue. His discussion of "equivalent weight" recalls an earlier one [THIS JOURNAL, **32**, 48 (1955)] by Meldrum.

Ramette [THIS JOURNAL, **37**, 344 (1960)] implies that this term has outlived its usefulness, saying "I grant the concepts of normality and equivalence no consideration whatsoever." Mellon [THIS JOURNAL, **38**, 49 (1961)] has also expressed disapproval of the normal system of specifying concentration of titrants.

While I am not out of sympathy with the idea of junking this term, I believe that its usefulness might be somewhat revived if a single simple definition covering all cases could be adduced in place of the hydra-headed traditional one.

I propose a new rule: to find the equivalent mass, divide the formula mass by the largest coefficient in the balanced equation, then multiply by the coefficient of the substance whose equivalent mass is desired.

As an example, consider the reaction $H_2SO_4 + 6HI = S + 3I_2 + 4H_2O$. Then (98)(1)/(6) = 16.3 grams of sulfuric acid will react with (128)(6)/(6) = 128 grams of hydrogen iodide to form (32)(1)/(6) = 5.3 grams of sulfur, (254)(3)/(6) = 127 grams of iodine, and (18)(4)/(6) = 12 grams of water. These are the equivalent masses of these substances by either definition, new or old.

The new rule will not always agree with the old definition, however. For instance, the old equivalent mass of each substance in the reaction $Zn + CuSO_4 = Cu + ZnSO_4$ is one-half the formula mass, but under the new rule the factor one-half does not appear. In practical use, this factor represents a needless complication, so that its elimination is a practical advantage of the new definition.

Lockwood [THIS JOURNAL, **38**, 326–329 (1961)] has emphasized that equivalent "weights" cannot be calculated without knowing the reaction to which they refer, nor can bottles be labeled with normality unless the intended use of the solution is known. Thus it is doubtful that the writing of balanced equations can be bypassed to a useful degree. In any event, application of the new rule to a part-equation balanced in the critical portions should allow all the freedom assignable to the older definition.

The new rule does not require differentiation between redox, metathetical, substitution, and Lewis acid-base reactions.

CRAYTON M. CRAWFORD

MISSISSIPPI STATE UNIVERSITY
STATE COLLEGE, MISSISSIPPI

To the Editor:

The article "Is There an Alternative to *p*H?" by Francis E. Crane, Jr. [J. CHEM. EDUC., **38**, 365 (1961)] included an excellent literature review and an interesting proposal. Like Crane, we recognize the difficulties associated with an understanding of *p*H by the layman and the beginning student. For example, our experience has shown that the average swimming pool maintenance man does not readily associate the number 7 with a neutral solution. As might be anticipated, the decrease or increase in *p*H values representing changes in acidity and alkalinity are seldom comprehended outside of the chemical laboratory.

In an attempt to prevent the recurrence of mistakes that are prevalent in swimming pool maintenance, we have devised another alternative scale as follows. [Acidity factor = (log $[H^+]$ + 7)14.3.]

Comparative Values for Representing Acidity

$[H^+]$	*p*H	Acidity factor
1×10^{0}	0	100
1×10^{-2}	2	71.5
$1 \times 10^{-3.5}$	3.5	50
1×10^{-5}	5	28.6
1×10^{-7}	7	0
1×10^{-9}	9	−28.6
$1 \times 10^{-10.5}$	10.5	−50
1×10^{-12}	12	−71.5
1×10^{-14}	14	−100

The acidity factor is zero for a neutral solution. Acidity factors for alkaline and negative solutions are negative and positive. Surveys conducted among groups representing swimming pool operators and corrosion engineers indicated a preference for the acidity scale over the *p*H scale.

Like Crane, we propose to supplement hydrogen ion activity and *p*H values, not to replace them. The acidity scale was developed primarily to provide a useful tool for those who fail to comprehend either *p*H or hydrogen ion activity.

RAYMOND B. SEYMOUR

SUL ROSS STATE COLLEGE
ALPINE, TEXAS

To the Editor:

R. Chandra, who wrote the article entitled "Non-Cyanide Detection of Cadmium in the Presence of Copper" [**38**, 409 (1961)], failed to indicate the chemistry involved in some of his procedures.

Procedure I, about which the author invited comments, is based on the soluble complex anion $[Cu(C_2O_4)]^{-2}$, which is formed with copper (II) in the presence of excess oxalate [H. SCHAEFER AND R. ABEGG, *Z. anorg. Chem.*, **45**, 293 (1905)]; H. L. RILEY, *J. Chem. Soc.*, **1929**, 1307; H. T. S. BRITTON AND M. E. D. JARRETT, *J. Chem. Soc.*, **1936**, 1489; and L. MEITES, *J. Am. Chem. Soc.*, **72**, 184 (1950), give quantitative data concerning this ion], and the equilibrium between the relatively insoluble $Cd(C_2O_4)$ [$K_{sp} = 1.44 \times 10^{-8}$, see W. C. VOSBURGH AND J. F. BECKMAN, *J. Am. Chem. Soc.*, **62**, 1028 (1940)] and a very weak complex anion $[Cd(C_2O_4)_2]^{-2}$. Instability constant with respect to CdC_2O_4 is about 0.034 according to Vosburgh and Beckman, *op. cit.* The low stability of the complex anion of cadmium causes most of the cadmium to remain precipitated even with excess oxalate.

With regard to Procedure II, copper is known to form complex anions with both carbonate and bicarbonate ions. On the basis of polarographic measurements, Meites (*op. cit.*) has suggested that these are $[Cu(CO_3)_3]^{-4}$ and $[Cu(HCO_3)_5]^{-3}$, respectively. The latter may also have a molecule of water coordinated to the copper or may actually be a carbonato complex, as carbonate ions are in equilibrium with the bicarbonate ions. A large negative charge would be built up, however, if all five are carbonate ions; therefore, the actual complex may be a mixed one. The carbonato complex is not of sufficient stability to overcome basic carbonate and hydroxide precipitation at higher *p*H's.

R. D. ARCHER

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RIVERSIDE, CALIFORNIA

To the Editor:

The intriguing article by Schultz and Sichels regarding the pyrolytic decomposition of carboxylate salts to ketones [THIS JOURNAL, **38**, 300 (1961)] raises the question as to the best laboratory method to use in preparing cyclopentanone from adipic acid. To answer this question, the following two experiments have been carried out.

In the first experiment, 14.6 g of powdered adipic acid was heated in a 125-ml distilling flask fitted with a thermometer which reached into the reaction mixture. The flask was heated with an open flame, and a reaction temperature of 315–330° was required before an appreciable amount of product distilled over. One and one-half hours was required to complete the reaction.

In the second experiment, the same amount of adipic acid was mixed with 1.6 g of $Ba(OH)_2 \cdot 8H_2O$ before the reaction was started. The reaction proceeded smoothly at 280–295° and was complete in 45 minutes.

Both reaction products were washed with 5 ml of water saturated with sodium bicarbonate and sodium chloride, and each was then dried over 1 g of calcium chloride. Each was distilled from a 10-ml distilling flask. The portion boiling between 126 and 132° was retained as cyclopentanone. The yield from the first experiment was 4.3 g while that from the second was 4.5 g.

While the difference in yields is not significant, the shorter reaction time and lower temperatures for the reaction in the presence of barium hydroxide suggests that the common laboratory preparation as given in many manuals is still the method of preference.

W. B. SMITH

TEXAS CHRISTIAN UNIVERSITY
FORT WORTH, TEXAS

Chem Ed

BOOK REVIEWS

Physical Chemistry

Farrington Daniels and *Robert A. Alberty*, both of the University of Wisconsin, Madison. 2nd Ed. John Wiley & Sons, Inc., New York, 1961. x + 744 pp. Figs. and tables. 16 × 23.5 cm. $8.75.

Although this book is called a second edition, actually it represents the tenth member of the noble family of Getman-Daniels-Alberty textbooks, which now spans nearly half a century. This rich tradition makes the present volume all the more remarkable in that it is a very real revision. Careful rewriting, imaginative reorganization, and addition of up-to-date material make it a truly new book.

Notable changes include three new chapters on Kinetic Theory, Spectroscopy, and Statistical Mechanics. Other chapters have been retitled, much material has been redistributed, and the order of presentation has been markedly changed. For example, the solid state has been relocated near the end of the book, abandoning the usual discussion of gases, liquids, and solids in sequence.

Viscosity, diffusion, and conductance are treated in a chapter entitled Irreversible Processes in Liquids. Surface tension is found in the chapter on Surface Chemistry. Other liquid properties such as dielectric constant and molar refraction are covered in the chapter on Molecular Structure, along with optical rotation. This section on optical rotation seems rather weak and adds little to the standard treatment to be found in most organic chemistry textbooks.

In the portions of the book devoted to classical thermodynamics, the reader will note a change of symbolism from F to G for the Gibb's free energy, to avoid confusion with the European use of F for the Helmholtz free energy. There is a good use of chemical potential and the partial molal Gibb's free energy throughout the book.

The all-important Clapeyron equation is better derived than formerly. Entropy is stressed more, and Trouton's rule is interpreted in terms of entropy of vaporization, and a comparison is made with entropies of fusion. The discussion of temperature is a bit unsatisfying in that it is never defined, but rather given as a concept, with emphasis on its measurement, instead of connecting it with the energy content of the system.

Both quantum and statistical mechanics have now properly become a definite part of undergraduate physical chemistry. Quantum mechanics is treated here at about the proper level with some enlargement of its former treatment to include more historical background and other topics. The important particle-in-a-box concept, necessary for the statistical treatment, is now included. The wave mechanics of the hydrogen atom and a discussion of orbitals is also new. There is an equally fine introduction to the importance and concepts of statistical mechanics. The Boltzman distribution among states, the partition function and its various forms and their relationship to the thermodynamic functions are all adequately discussed.

The chapter on Spectroscopy is a welcome one. It surveys the various aspects of this field, including the microwave region and electron magnetic resonance. The examples and problems here will be found especially helpful.

The most outstanding feature of the text as in the earlier edition, is the generous presentation of tables, complete with references. These serve not only to illustrate and confirm principles and equations, but to give the entire course a solid foundation in experimental-deductive logic, essential to the spirit of physical chemistry.

The book is well-supplied with examples, references, and problems, some with answers. Illustrations are adequate, although a few more three-dimensional figures would add to the attractiveness and clarity of the book, particularly in certain phase diagrams. There is a most useful appendix discussing various subjects from mathematical procedure to Eyring's absolute rate theory. All in all, this book is a stimulating, well-written textbook that will please both instructor and student.

TOM R. THOMSON
Arizona State University
Tempe

Standard Methods of Clinical Chemistry. Volume 3

Edited by *David Seligson*, Yale University School of Medicine, New Haven, Connecticut. Academic Press, Inc., New York, 1961. x + 230 pp. Figs. and tables. 16 × 23.5 cm. $6.50.

The current volume in this series maintains the high standards established by the previous ones. The organization, style, and typography make for thoroughness, clarity, and ease of reading and the methods described have been checked for accuracy and precision as well as for their significance as standard, or reference, procedures.

Although the book is obviously of greatest use to practicing clinical chemists it should be of interest to instructors of courses which include even a modicum of clinical chemistry. The example typified by the statement of the editor in the Preface should be of value in any course of analytical methods: "A current temptation exists, for the less skilled chemists, to rely on specialized instruments, 'test kits' prepared reagents, and sometimes secret commercial formulations because these can be bought easily. Very few good chemists perform analyses with solutions made outside their own laboratories. Sometimes, the secret to successful analyses lies in the skillful preparation of reagents. The methods in this volume clearly and carefully instruct the reader on preparation of standards and reagents."

As usual, each method includes the historical background, the principles involved, the details of the procurement, preparation and/or standardization of all necessary reagents, step-by-step proce-

Reviewed in This Issue

Farrington Daniels and *Robert A. Alberty*, Physical Chemistry

David Seligson, Editor, Standard Methods of Clinical Chemistry. Volume 3

V. V. Cherdyntsev, Abundance of Chemical Elements

A. J. Swallow, Radiation Chemistry of Organic Compounds

Carl Mahr, Anorganisches Grundpraktikum

C. G. Cannon, Editor, Electronics for Spectroscopists

Omer K. Whipple, Chemical Properties and Identification of Ions: An Introduction to Semimicro Qualitative Analysis

A. V. Tobolsky, Properties and Structure of Polymers

A. I. Busev and *N. G. Polianskii*, The Use of Organic Reagents in Inorganic Analysis

C. P. Stewart and *A. Stolman*, Editors, Toxicology: Mechanisms and Analytical Methods. Volume I

F. Albert Cotton, Editor, Progress in Inorganic Chemistry. Volume 2

Arthur Osol and *Robertson Pratt*, Editors, The Dispensatory of the United States of America. New Drug Developments Volume

dures with explanatory notes regarding precautions needed to attain high precision and accuracy, all calculations, and concise discussions of the physiological, clinical, and, where appropriate, forensic or genetic significance of the results. All methods have been checked in one or two independent laboratories; the modifications suggested by the checkers are included. An extensive list of general and specific references is given at the end of each chapter and complete subject and author indices at the end of the volume. Several novel pieces of equipment are described, the most noteworthy being a "chloridimeter."

More emphasis is placed in this volume on methods of use in toxicology or forensic medicine. The chapters in which these methods are described also include rather thorough discussions of the pharmacology and toxicology of the drugs or toxic agents.

Methods are given for the determination of: alcohol in biological materials, aldolase, arsenic in biological materials, ascorbic acid in blood and urine, barbiturates, catechol amines, chloride, cholinesterase in red cells and plasma, creatine and creatinine in blood and urine, fibrinogen, gastric acidity, hemoglobins, hydrocortisone in plasma, lactic acid, mercury in urine, phosphohexose isomerase, salicylates, sulfonamides, and transaminase. Three of these require special or expensive equipment, such as that required for boundary electrophoresis.

GORDON H. PRITHAM
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University Park, Pennsylvania

Abundance of Chemical Elements

V. V. Cherdyntsev, S. M. Kirov State University, Alma-Ata, U.S.S.R. Translated by *Walter Nickiporuk*, California Institute of Technology, Pasadena. University of Chicago Press, Chicago, Illinois, 1961. x + 304 pp. Figs. and tables. 16 × 24 cm. $10.

In the words of the author, "This book is neither a textbook nor a work of reference. My aim was to give an account of the vast and varied material on the abundance of the chemical elements. In this objective I was guided by fundamental ideas on the stability of atomic nuclei and on their formation in a neutron environment." In spite of the author's comments, this book should find utility in an academic library both as an upper division undergraduate and graduate text for special topics courses in chemistry and as a handy reference work. The material discussed lies in a very interesting interdisciplinary field, that includes aspects of physics, astrophysics, geophysics, geology, geochemistry, and chemistry.

The book was first published in Moscow in 1956 and has been revised by the author through the first half of 1958 for the American edition. The original Russian text has been very capably translated into English by Walter Nichiporuk of Cal Tech. The quality of the translation, the printing, and the general appearance of this book are in this reviewer's opinion, far superior to many similar translations of Russian works which have appeared recently. The translator has greatly enhanced the value of this book as a reference source by annotating comments, criticisms, and references to recent experimental data up to the end of 1960 in the form of footnotes. In some cases, these footnotes exceed one-third the length of a printed page and it is unfortunate that this material could not have been incorporated into the text itself, through additional consultations with the author. It is somewhat disconcerting to study a table of data supporting a point made by the author and then discover a footnote refering to recent work (in some cases only the literature citation, but not the specific data, is given) which might invalidate the author's conclusion. The updating by means of footnotes is also not entirely uniform. For example, reference is made by the translator in a footnote of page 52 to the recent discovery of enrichment of xenon isotopes in chondritic meteorites. However, the author's statements on page 216, "... xenon isolated from stony meteorites does not show an excess content of Xe^{129}." and, "The present isotopic composition of xenon, which is the same for the earth and meteorites, ..." stand in the text with no annotation.

The book is divided into three major chapters: I, The abundance of the chemical elements in the earth's crust and the cosmic systems; II, The elementary theory of stability of atomic nuclei; III, Regularities in the abundance of isotopes and the problem of the formation of atomic nuclei. Many of the author's own investigations on the occurrence and distribution of the elements are discussed. Emphasis is placed on the abundances in the earth's crust, and the role of radioactive decay in the establishment of the present elemental abundances is discussed at length.

This volume is unique in that it is devoted to a survey of elemental abundances from the viewpoint of the stability of the atomic nuclei, using new data in many fields that previously were obtained only by an extensive literature search. As one of the few books available in this important new interdisciplinary field, it should find utility in the library of the advanced student and the researcher, as well.

WILLIAM D. EHMANN
University of Kentucky
Lexington

Radiation Chemistry of Organic Compounds

A. J. Swallow, Imperial College of Science and Technology, London, England. Pergamon Press, Inc., New York, 1960. ix + 380 pp. Figs. and tables. 15 × 22 cm. $15.

This book is distinguished by a remarkably thorough coverage of the pertinent literature up to the beginning of 1959. Its best features are the "Summary and Conclusion" which exist at the end of each chapter and the "Conclusions" at the end of the book. For many, these portions will provide ideas for future work. It is no exaggeration to say that every serious worker in the field of radiation chemistry will profit by personal ownership of a copy of this volume.

The choice and order of subject matter (general introduction, experimental methods, water and aqueous systems, aliphatics, aromatics, polymers, dyestuffs, substances of biological interest, and significance for other fields) are excellent so that the book is an important reference work—particularly to obscure references.

Unfortunate features do, however, limit its usefulness and readability.

The presentation is essentially noncritical. For many purposes coverage is more important than criticality. Nevertheless, style and flavor of presentation are a cause of trial and inconvenience for the informed reader and may be very misleading for the novice. Hardly ever, if at all, in the text is there a reference to the name of a contributor. Thus, all ideas, all "facts" appear equally weighted—without regard not only to the reliability of the author of a particular statement but also to the objective of the work from which a particular "statement of fact" is drawn. The difficulty is compounded by the method of presentation of references, which appear in code in the text and as a bibliographical unit in the rear of the book. Doubtless, such arrangement does lower costs for the publisher but the total effect is to make the book, as a book, almost unreadable. Anyone who pays $15 for a 380-page book has the right to demand greater convenience as well as a book which will not be destroyed by abnormal and repeated turning from text to bibliography to text and back again.

Of course, a text like this has some fundamental faults, including statements which are not good and, in some cases, utterly wrong. A statement of the "first law of radiation chemistry" is pompous and certainly incorrect for one area of the subject. The principal fault, perhaps, is that the text assumes simplicity of correlation between radiation chemistry and other branches of reaction kinetics; there appears to be no awareness that many of the fundamental notions of mechanism are presently being subjected to critical revaluation by those interested in matters of theory. In the beginning of this review, I said that many will receive ideas for future work from a reading of the "Conclusions." The qualifying statement must also be made that many such ideas may not be good and may direct the unwary research worker into areas of triviality.

MILTON BURTON
University of Notre Dame
Notre Dame, Indiana

(*Continued on page A916*)

Jan.....1–50, AI–A62	July....333–380, A453–A506
Feb.....51–104, A67–A126	Aug.....381–432, A511–A558
Mar....105–158, A131–A216	Sept....433–482, A563–A704
Apr....159–222, A221–A296	Oct.....483–536, A709–A780
May....223–276, A301–A380	Nov.....537–588, A785–A860
June....277–332, A385–A448	Dec.....589–656, A865–A934

ANNUAL INDEX
Journal of Chemical Education
Volume 38, 1961

The annual index for Volume 38 of the Journal of Chemical Education is divided into several parts. Authors are listed in three sections: authors of articles, books reviewed, and letters to the editor. The subject index includes both subjects and titles to articles; the books reviewed are listed in a separate section. The principal subject entries are distinguished from titles by printing the former in italics. Portraits, including medals, are indicated by (P), letters to the editor by (L), errata by (E).

AUTHOR INDEX

ANDERS, EDWARD, AND GOLES, GORDON G. Theories on the origin of meteorites... 58
ANDERSON, ROBBIN C.—*See* PINO, LEWIS N.
ATKINSON, G. F., AND MCBRYDE, W. A. E. Graphical representation of hydrolysis of the ferric ion..... 127

BACON, E. K. A pioneer summer institute: The G.E. Science Fellowship Program at Union College..... 39
BAILAR, JR., JOHN C. Chemical education—then and now..... 434
BAKER, PHILIP S. A rapid efficient method for reducing certain metallic isotopic oxides..... 117
BAKER, WILBUR L. Models illustrating types of orbitals and bonding..... 606
BANKS, JAMES E. The equilibria of complex formation. Textbook errors, 32.. 391
BARLOW, GRANT H. Paper electrophoretic study of rates of consecutive reactions: the deamidation of vitamin B_{12}..... 37
BARNARD, ROBERT, AND WOODRIFF, RAY. A low cost direct current source for the laboratory..... 521
BASSHAM, J. A. New aspects of photosynthesis..... 151
BENTLEY, K. E.—*See* MARTIN, E. L.
BESTE, LAWRENCE F. A Geometric approach to the Gibbs-Duhem equation. 509
BJORK, R. G.—*See* STOCK, JOHN T.
BORYTA, DANIEL A.—*See* MARKOWITZ, MEYER M.
BOTDORF, RUTH. Using filmed demonstrations in general chemistry..... 95
BOTTEI, RUDOLPH S., AND GERACE, PAUL L. A thermobalance for student experiments..... 568
BRAUNSTEIN, JERRY, AND YOUNG, RAYMOND H. Measurement of the half life of UX_2(Pa-234) with a dipping counter.. 31
BREWER, LEO. Non-stoichiometry in nonmetallic compounds..... 90
BRIGGS, THOMAS S.—*See* OBOLD, JOSEPH K.
BROOKER, ROBERT M., AND RAPALA, RICHARD T. The National Science Fair-International and the ACS Chemistry Awards..... 209
BROWN, HERBERT C. New selective reducing agents..... 173
BRUMLIK, GEORGE C. Molecular models featuring molecular orbitals..... 502
BULL, H., III—*See* SICILIO, F.
BUNNETT, JOSEPH F. The chemistry of benzyne..... 278
BURGESS, T. E., LOOBY, J. T., AND ORR, W. C. Disposable plastic mounts for radioactive samples..... 208
BURNS, ROBERT L., AND GRUEN, FRED M. Benzenoid nitro-compounds as organic reagents for tin(II)..... 410
BURTON, FREDERICK—*See* WILLIAMS, T. R.
BUTLER, JAMES N. An approach to complex equilibrium problems..... 141
Calculating molar solubilities from equilibrium constants..... 460

CAMPBELL, J. A. The chemical education materials study..... 2
The paddle-wheel Crookes tube. Textbook errors, 33..... 480
CAPRIOLA, GEORGE—*See* MARKOWITZ, MEYER M.
CARMODY, WALTER R. An easily prepared wide range buffer series..... 559
CARSWELL, D. J. Convenient radioactivity sources..... 352
CASSIDY, HAROLD G. The problem of the sciences and the humanities..... 441
CERKOVNIKOV, E. Alekxei Evgen'evich Chichibabin..... 622
CHALMERS, DAVID G.—*See* MANES, MILTON
CHAMPEAUX, VERNEL C.—*See* MANN, CHARLES K.
CHAN, ALFRED, JR. A simple test for nitric acid fumes..... 412
CHANDRA, RAMESH. Detection of phosphate in the presence of arsenate..... 408
Non-cyanide detection of cadmium in the presence of copper..... 409
Rapid detection of cations of copper group..... 413
CHOI, IL C.—*See* OSBURN, JAMES O.
CLAPP, L. B.—*See* STRONG, L. E.
—*See* WILLEFORD, BENNETT R.
CLARK, ROY W., AND SELBIN, JOEL. Coordination chemistry in general chemistry texts..... 466
CLEVER, H. LAWRENCE. Heat of precipitation: A general chemistry experiment 470
COHEN, IRWIN. Moles and equivalents: Quantities of matter..... 555
The shape of the 2p and related orbitals. Textbook errors, 28..... 20
COLBURN, CHARLES B. Recent developments in N—F chemistry..... 180
COLTHUP, NORMAN B. Vibrating molecular models: Frequency shifts in strained ring double bonds..... 394
CONNOLLY, J. W.—*See* RADELL, JACK
COOK, W. B.—*See* GARRETT, A. B.
COPLEY, GEORGE NOVELLO. The mole in quantitative chemistry..... 551
CORMACK, JOAN M.—*See* MARIELLA, RAYMOND P.
CORYELL, CHARLES D. The chemistry of creation of the heavy elements..... 67
COTTRILL, SHARON L.—*See* TOBEY, S. W.
CRANE, FRANCIS E., JR. Is there an alternative to pH?..... 365
CROCKER, R. E.—*See* KAPLAN, M. L.
CROWELL, D. C., SISTER AGNITA—*See* MARTINETTE, B. V. M., SISTER MARY
CURET, J. D.—*See* ROIG, E.

DAILEY, G. W., HUFF, R. B., KANO, J., QUEEN, L. D., and PATTERSON, C. S. Isopiestic vapor pressure apparatus... 28
DARLING, S. F. Demonstrating enzymatic hydrolysis of glucosides by paper chromatography..... 508
DE MENT, JACK. Translating and rejargonising the little-languages of science..... 415
DIRKSE, THEDFORD P. Textbook errors, 30. The ionization constant of water. 260
DRESSEL, JUTTA (RALPH E. OESPER, *Translator*). The discovery of Germanin by Oskar Dressel and Richard Kothe.... 620
DUKE, FREDERICK R. Oxidation-reduction mechanisms..... 161
DUNICZ, BOLESLAW LUDWIK. Surface area of activated charcoal by Langmuir adsorption isotherm..... 357
DUNLAP, W. CRAWFORD. Recent developments in semiconductors..... 238
DUTTON, FREDERIC B., *Editor*. Tested demonstrations. A23, A99, A179, A289, A357, A419, A491, A545, A637, A751, A813, A895
DUVEEN, DENIS I. Personalized bibliography: An approach to the history of chemistry..... 418

EDWARDS, J. O.—*See* STRONG, L. E.
EHMANN, WILLIAM D. Recent improvement in our knowledge of cosmic abundances..... 53
ETZEL, HOWARD W. Ionic crystals..... 225
EVERS, E. CHARLES. The nature of metal-in-amine solutions..... 590
EYRING, HENRY—*See* JONES, LEON L.

FERNELIUS, W. CONARD—*See* MANCH, W.
FERRARO, JOHN R. Inorganic infrared spectroscopy..... 201

Ferry, John D. Rheology in the world of neglected dimensions..... 110
Fill, M. A.—*See* Stock, John T.
Fletcher, Edward A. Total and static thermodynamic properties: Old gas dynamic properties which may be new to the chemist..... 257
Forchheimer, Otto L., Newkirk, Terry F. Recent advances in solid state inorganic materials..... 183

Gaddis, Shirley W. A simple filtration technique for gravimetric determinations..... 5
Garrett, A. B., Cook, W. B., and Lippincott, W. T. College chemistry course for non-science majors: A report of the Montana State Conference..... 253
Garvin, James E. Student experiment with filter paper electrophoresis..... 36
Gerace, Paul L.—*See* Bottei, Rudolph S.
Gill, S. J. A demonstration of optical rotation with an "Eskimo Yo-Yo".... 263
Gillis, J. Kekulé's life at Ghent (1858–1867). (Oesper, Ralph E., *Translator*)..... 118
Goles, Gordon G.—*See* Anders, Edward
Gordon, Louis, and Salesin, Eugene D. Precipitation from homogeneous solution: A lecture demonstration..... 16
Gorman, Mel. Antecedents to modern concepts of configurational symmetry in chemistry..... 99
Graves, Carlton S. Melting point apparatus..... 634
Gregg, Donald C. Asking students to use facts..... 478
Gruen, Fred M.—*See* Burns, Robert L.
Guggenheim, E. A. The mole and related quantities..... 86, (E)131, (L)554

Haiduc, I. A systematization of inorganic cyclic compounds..... 134
Hakala, Reino W. A new derivation of the Boltzman distribution law..... 33
Hall, H. Tracy The synthesis of diamond..... 484
Hall, M.—*See* Thiessen, G. W.
Halliwell, H. F.—*See* Nyburg, S. C.
Hanson, Marvin Wayne. Chloride, bromide, and iodide determination.... 412
Harris, Daniel L. A rapid, direct-reading micro-balance..... 469
Harris, E. Lyndol—*See* Vick, Maurice M.
Head, William F., Jr., and Marsh, Max M. An inexpensive coulometer for titrations with externally generated reagents..... 361
Hein, George E. The Liebig-Pasteur controversy: Vitality without vitalism 614
Hepler, Loren G.—*See* O'Hara, William F.
Hoare, James P. A simple method for finding slopes..... 570
Hollingsworth, C. A.—*See* Smith, George B.
Honig, J. M. Elements of order-disorder theory: Applications to gas adsorption, binary solutions, alloys, and ferromagnetism..... 538
Hoover, William G. and Shriver, Duward. Inexpensive Stuart-type molecular models..... 295
Hornyak, Frederick M. A flashbulb bomb calorimeter..... 97
Huff, R. B.—*See* Dailey, G. W.
Huisgen, Rolf. Richard Willstätter..... 10
Hurd, Charles D. The general philosophy of organic nomenclature..... 43

Inde, Aaron J. Editorially speaking..... 483
The Karlsruhe Congress: A centennial retrospect..... 83
Innes, K. Keith. Viscosities of butanol-heptane mixtures..... 564

Jean, Marcel. Substitute for separatory funnels..... 515
Joiner, Curtis R. Pesticide residue control under the Food, Drug, and Cosmetic Act..... 370
Jones, Carl T., and Williams, Max B. A new qualitative reagent for aluminum 411
Jones, Gurnos. The Markovnikov rule.. 297
Jones, Leon L., and Eyring, Henry. A model for optical rotation..... 601

Kadis, Barney—*See* Scher, Irwin
Kang, J.—*See* Dailey, G. W.
Kaplan, M. L., Crocker, R. E., and Sargent, Herbert. A teflon bearing for stirrers..... 577
Katz, Lewis. A systematic way to avoid Le Chatelier's principle in chemical reactions..... 375
Kerker, M.—*See* Matijevic, E.
Ketcham, Roger, and Low-Beer, Anne de G. A simple test for the detection of phosphorus in organic compounds.. 414
Kirschner, Stanley. Adapting machine-sorted punched card systems to hand sorting and coding..... 526
Knight, J. A.—*See* Sicilio, F.
Knox, Bruce E., and Palmer, Howard B. The uses and abuses of bond energies. 292
Kohman, Truman P. Introduction: Cosmochemistry symposium..... 52
Chronology of nucleo-synthesis and extinct natural radioactivity..... 73
Komuro, Yasuyuki, and Sôné, Kozo. Paper-made crystal models..... 580
Kratohvil, J. P.—*See* Matijevic, E.
Kremer, Chester B., and Wilen, Samuel H. Apparatus for the Friedel-Crafts reaction..... 306
—*See* Wilen, Samuel H.

Lamb, J. F.—*See* Perkins, Gerald, Jr.
Langer, Stanley H., and Pantages, Peter. A convenient safety shield for small-scale apparatus..... 634
Lansbury, Peter T. A convenient method for demonstration of stable carbanions..... 307
Lee, Shiu. A redefinition of "mole"..... 549
Leegwater, Arie—*See* Wolthuis, Enno
Leisten, J. A. Experiments in acid-base catalysis..... 132
A group experiment on the Hammett sigma-rho relation..... 302
Lewin, S. Z. Chemical instrumentation.. A5, A71, A135, A225, A305, A389, A457, A515, A567, A713, A789, A000
Lippincott, W. T. Report from the Committee on Institutes and Conferences, Division of Chemical Education, ACS(P)..... 529
—*See* Garrett, A. B.
Lockwood, Karl L. Redox revisited.... 326
Looby, J. T.—*See* Burgess, T. E.
Low-Beer, Anne de G.—*See* Ketcham, Roger

MacNevin, William. Analytical chemistry and the non-analytical graduate student..... 527
A laboratory practical examination in quantitative chemistry..... 144
Macomber, Robert D. Chemistry accidents in high school..... 367
MacWood, George E., and Verhoek, Frank H. How can you tell whether a reaction will occur?..... 334
Mahr, Carl. Preparative separations replace typical qualitative analysis..... 407
Malinin, Dorothy R., and Yoe, John H. Development of the laws of colorimetry 129
Manch, W., and Fernelius, W. Conard. The structure and spectra of nickel(II) and copper(II) complexes..... 192
Manes, Milton, and Chalmers, David G. Laboratory heating bath using fluidised solids..... 102
Mann, Charles K., and Champeaux, Vernel C. A constant current source for student use..... 519
Mariella, Raymond P., and Cormack, Joan M. Backgrounds of graduate school chemistry faculties..... 427
Markowitz, Meyer M., Boryta, Daniel A., and Capriola, George. A motor-driven variable transformer..... 96
Marsh, Max M.—*See* Head, William F., Jr.
Martin, E. L., and Bentley, K. E. Apparatus for oxygen-sensitive volumetric solutions..... 264
Martin, J. C. NMR spectroscopy as an analytical tool in organic chemistry... 286
Martinette, Sister Mary, B.V.M., and Crowell, Sister Agnita, D.C. Adapting hospital "castoffs" for use in a chemistry laboratory..... 619
Masterton, W. L. Analysis of an aluminum-zinc alloy..... 558
Matijevic, E., Kratohvil, J. P., and Kerker, M. A simple demonstration of some precipitation and solubility effects..... 397
Mauer, L. E.—*See* Perkins, Gerald, Jr.
McBryde, W. A. E.—*See* Atkinson, G. F.
McDaniel, Darl H.—*See* Smith, George B.
McGregor, S. D.—*See* Tobey, S. W
McKetta, John J., and Schechter, Robert S. A flexible undergraduate chemical engineering program..... 261
McMillan, Juan A. Electron paramagnetic resonance of free radicals..... 438
Michels, William. A simple method for cleaning buret tips..... 548
Mikulak, Robert, and Runquist, Olaf. Molecular weights by cryoscopy..... 557
Miles, Wyndham D. Robert Maskell Patterson: First to teach atomic theory in America?..... 561
Miller, G. H.—*See* Pritchard, G. O.
Moore, Walter J. Diffusion in solids and the gaseous corrosion of metals... 232
Morrell, William E. Summer institutes: Review and future plans..... 448
Moulton, Wilbur N. Textbook errors, 34. Dipole moments of symmetrical organic molecules..... 522
Mucci, J. F., Spiegel, Dorothy E., and Stearns, Robert L. Ion exchange-radiochemical experiment..... 348
—*See* Stearns, Robert L.
Myers, Howard M. Revised teaching of biochemistry..... 363
Mysels, Karol J. Textbook errors, 35. Miscellanea No. 4: Chromyl chloride test for chlorides; "monovalent oxygen" compounds; ternary diagrams.. 627
Editor, Textbook errors....20, 138, 260, 300, 391, 480, 522, 627

Nechamkin, Howard. The course content of general chemistry..... 255
Neville, Roy G. "The Sceptical Chymist," 1661. A tercentenary tribute..... 106
Newkirk, Terry F.—*See* Forchheimer, Otto L.
Nishijima, Yasunori, and Oster, Gerald. Diffusion under the microscope..... 114
Noyce, William K. Stereoisomerism of carbon compounds..... 23
Nyburg, S. C., and Halliwell, H. F. A geometric approach to extensive properties..... 123, (E)329, (L)329

Obold, Joseph K., and Briggs, Thomas S. A summer chemistry program for teenagers..... 47
Oesper, Ralph E.—*See* Dressel, Jutta
—*See* Gillis, J.
—*See* Timmermans, Jean
O'Hara, Williams F., Wu, Ching-Hsien, and Hepler, Loren G. Temperature and power measurements in precision solution calorimetry..... 512

O'Reilly, D. E. Electronic configuration of metal oxides 312
Orr, W. C.—*See* Burgess, T. E.
Osburn, James O. Analog computer instruction: A plugboard teaching aid .. 492
—and Choi, Il C. Float method for the analysis of solutions 578
Oster, Gerald—*See* Nishijima, Yasunori
Overberger, C. G.—*See* Riley, Reed F.

Palmer, Howard B.—*See* Knox, Bruce E.
Palmer, R. C.—*See* Sicilio, F.
Pantages, Peter—*See* Langer, Stanley H.
Parry, R. W. Introduction. Symposium: Recent advances in inorganic chemistry 160
Patterson, C. W.—*See* Dailey, G. W.
Pearson, Ralph G. Crystal field theory and substitution reactions of metal ions 164
Pecsok, Robert L. Gas chromatography. 212
Perkins, Gerald, Jr., Wimberley, J. W., Lamb, J. F., and Maurer, L. E. The turbidimetric determination of lead ... 358
Peterson, M. D.—*See* Sicilio, Fred
Phillips, J. P.—*See* Thomas, Alan T.
Pino, Lewis N., and Anderson, Robbin C. Institutes in the academic year ... 451
Ploeg, John Vander—*See* Wolthuis, Enno
Podall, Harold E. Recent developments in metal carbonyl synthesis 187
Pritchard, G. O., and Miller, G. H. Safety device for vacuum diffusion pumps 525
Proctor, James S., and Roberts, John E. Analysis of aspirin: A conductometric titration 471

Queen, L. D.—*See* Dailey, G. W.

Radell, Jack, and Connolly, J. W. Fractionation technique for vacuum distillation 459
Radin, Norman S. Incorporating radioisotope techniques into the chemistry curriculum 344
Ramsey, J. B. The basis for a new conception of an electrode potential 353
Rapala, Richard T.—*See* Brooker, Robert M.
Ratnam, C. V. Depsides: A synthesis by Emil Fischer 93
Raw, C. J. G. Chemical equilibria in imperfect gases 140
Reinmuth, W. H. Three-dimensional representation of electrodeposition 149
Rieckehoff, I. G.—*See* Roig, E.
Rieman, William, III. Salting-out chromatography: A review 338
Riley, Reed F., and Overberger, C. G. A summer research participation program for high school students 424
Ritchie, A. S. A paper chromatographic scheme for the identification of metallic ions 400
Roberts, John E.—*See* Proctor, James S.
Robertson, G. Ross. New fundamental designs for academic chemistry buildings 474
Rogers, L. B. Contemporary analysis and the chemistry curriculum 455
Roig, E., Rieckehoff, I. G., Russo, C. S., and Curet, J. D. Radioisotope demonstration of common ion effect on solubility 350
Roth, Jay S. Ribonucleic acid and protein synthesis 217
Runquist, Olaf—*See* Mikulak, Robert
Russo, C. S.—*See* Roig, E.

Salesin, Eugene D.—*See* Gordon, Louis
Sargent, Herbert—*See* Kaplan, M. L.
Schaefer, William P.—*See* Swift, Ernest H.
Schechter, Robert S.—*See* McKetta, John J.
Scher, Irwin, Walsh, Edward, and Kadis, Barney. A biochemistry laboratory experiment 632
Schmuckler, Joseph S., and Siegelman, Irwin. The chemistry student and the chemical industry 148
—*See* Siegelman, Irwin
Schufle, J. A. Deionized water by electrodialysis 17
Schultz, Harry P., and Sichels, Jackson P. The pyrolytic decomposition of carboxylate salts to ketones. Textbook errors, 31 300
Scott, Allen B. Introduction: Chemistry of the solid state symposium 224
Electrochemistry of the solid state 317
Selbin, Joel—*See* Clark, Roy W.
Shapiro, Samuel. Device to convert a hypodermic syringe into a multi-purpose apparatus 626
Sherr, Allan E. Educational activities of the Western Connecticut ACS Section 428
Shriver, Duward—*See* Hoover, William G.
Sichels, Jackson P.—*See* Schultz, Harry P.
Sicilio, F., Bull, H., III, Palmer, R. C., and Knight, J. A. An inexpensive versatile multi-column gas chromatograph for students 506
—and Peterson, M. D. Ratio errors in pseudo first order reactions 576
Siebring, B. R. Institutional influences in the graduate training of eminent chemists 630
Siegel, Bernard. Hydride formation by atomic hydrogen reactions 496
Siegelman, Irwin, and Schmuckler, Joseph S. The Franklin Institute chemistry exhibit: A design for learning 597
—*See* Schmuckler, Joseph S.
Smith, George B., Hollingsworth, C. A., and McDaniel, Darl H. Solvation and association from colligative properties by graphical means 489
Smith, Jonathan S., II—*See* Spialter, Leonard
Smith, Julian F. Abbreviations in Russian chemical literature 41
Smith, S. G., and Stevens, I. D. R. Nomographs for first and second order kinetics 574
Sone, Kôzô—*See* Komuro, Yasuyuki
Spialter, Leonard, and Smith, Jonathan S., II. Dye solutions for the preparation of colored slides 473
Spiegel, Dorothy E.—*See* Mucci, J. F.
Sporer, A. H. The role of chemistry in computers 323
Stearns, Robert L., and Mucci, Joseph F. Using the decay of Ba-137 to determine resolving time in G-M counting ..... 29, (L)329
—*See* Mucci, J. F.
Stevens, I. D. R.—*See* Smith, S. G.
Stewart, Ross. The reactive intermediates of organic chemistry 308
Stock, John T. A titration controller for demonstrating the behavior of acid-base indicators 633
—Fill, M. A., and Bjork, R. G. Device for charging micro-tubes with toxic reagents 524
Strong, F. C. Faraday's laws in one equation 98
Strong, L. E., Clapp, L. B., and Edwards, J. O. Geometry in the beginning chemistry course 530

Swift, Ernest H., and Schaefer, William P. A qualitative elemental analysis system correlated with the periodic table 607

Taylor, John K.—*See* Young, Jay A.
Thiessen, G. W., and Hall, M. An improved gas generator for elementary chemistry 19
Thomas, Alan T., and Phillips, J. P. Ion exchange paper chromatography .. 406
Thomas, John M. The existence of endothermic adsorption. Textbook errors, 29 138
Timmermans, Jean (Oesper, Ralph E., *Translator*). Walther Spring (1848–1911) 422
Frederic Swarts (1866–1940) 423
Tobey, Stephen W. Constant current supply for electrokinetic experiments .. 517
Simplified moving-boundary transference cell 516
—McGregor, S. D., and Cottrill, Sharon L. Determination of saponification equivalents of phenolic esters. 611

Vasilakes, William. Solid sample identification analysis in high school chemistry 146
Verhoek, Frank H.—*See* MacWood, George E.
Vick, Maurice M., and Harris E. Lyndol. Ion exchange separation of cadmium 414
Vikin, J. Chemical information centers of Latin America 628

Walsh, Edward—*See* Scher, Irwin
Waltcher, Irving—*See* Wilen, Samuel
Wendlandt, Wesley W. A controlled atmosphere thermobalance 566
Reaction kinetics by differential thermal analysis 571
Weisfeld, L. B. The catalysed reaction of phenyl isocyanate with butanol. A thermoanalytical kinetic experiment .. 88
Weissbart, Joseph. Fuel cells—electrochemical converters of chemical to electrical energy 267
Wilen, Samuel H., Kremer, Chester B., and Waltcher, Irving. Polystyrene—a multistep synthesis 304
—*See* Kremer, Chester B.
Willeford, Bennett R., Jr., and Clapp, Leallyn B. The undergraduate training of chemistry majors: A report of the Bucknell Conference 251
Williams, Ferd E. The luminescence of inorganic crystals 242
Williams, Max B.—*See* Jones, Carl T.
Williams, T. R., and Burton, Frederick. A specific qualitative reagent for copper 413
Wimberley, J. W.—*See* Perkins, Gerald, Jr.
Wolthuis, Enno, Leegwater. Arie, and Ploeg, John Vander. Heat of reaction and H_2SO_4 concentration 472
Woodriff, Ray—*See* Barnard, Robert
Wu, Ching-Hsien—*See* O'Hara, William F.

Yoe, John H.—*See* Malinin, Dorothy R.
Young, Jay A. Programmed instruction in chemistry: An invitation to participate 463
—and Taylor, John K. Chemical projects: Research ideas for young chemists 6, 8–9, A103-4, A183-4, A263-4, A353-4, A421-2, A501-2, A555-6, A611-2, A757-8, A819-20, A891-2
Young, Raymond H.—*See* Braunstein, Jerry

* * *

SUBJECT INDEX

ACS Chemistry Awards, The National Science Fair-International and the. Robert M. Brooker and Richard T. Rapala 209
ACS Section, Western Connecticut, Educational activities of the. Allan E. Sherr 428
Abbreviations in Russian chemical literature. Julian F. Smith 41
Abundances, cosmic, Recent improvement in our knowledge of. William D. Ehmann 53
Academic year, Institutes in the: Review and future plans. Lewis N. Pino and Robbin C. Anderson 451
Accidents, Chemistry, in high school. Robert D. Macomber 367
Acid-base catalysis, Experiments in. J. A. Leisten 132
Acid-base chemistry, Lewis, A reaction to introduce. Tested demonstration A491
Acid-base indicators, A titration controller for demonstrating the behavior of. John T. Stock 633
Adapting hospital "castoffs" for use in a chemistry laboratory. Sister Mary Martinette, B.V.M., and Sister Agnita Crowell, D.C. 619
Adapting machine-sorted punched card systems to hand sorting and coding. Stanley Kirschner 526
Adsorption, endothermic, The existence of. Textbook errors, 29. John M. Thomas 138
Adsorption, gas, binary solutions, alloys, and ferromagnetism, Applications to. Elements of order-disorder theory. J. M. Honig 538
Adsorption isotherm, Langmuir, Surface area of activated charcoal by. Bolesław Ludwik Dunicz 357
Alekseĭ Evgen'evich Chichibabin. E. Cerkovnikov 622
Alloy, aluminum-zinc, Analysis of. W. L. Masterton 558
Alloys, gas adsorption, binary solutions, and ferromagnetism, Applications to. Elements of order-disorder theory. J. M. Honig 538
Aluminum, A new qualitative reagent for. Carl T. Jones and Max B. Williams. 411
Aluminum-zinc alloy, Analysis of an. W. L. Masterton 558
Ammonia, Catalytic oxidation of. Tested demonstration A23
Analog computer instruction: A plugboard teaching aid. James O. Osburn 492
Analysis, contemporary, and the chemistry curriculum. L. B. Rogers 455
Analysis, identification, Solid sample, in high school chemistry. William Vasilakes 146
Analysis of an aluminum-zinc alloy. W. L. Masterton 558
Analysis of aspirin: A conductometric titration. James S. Proctor and John E. Roberts 471
Analysis of solutions. Float method for the. James O. Osburn and Il C. Choi 578
Analytical chemistry
Alternative to pH? ..... 365, (L)638
Apparatus for solution dispensing 264
Buffer series 559
Chromatography A515, A567, A713, A789
Complex equilibria 141
Complexation equilibria 391
Density by float method 578
Elemental analysis 607
Gas chromatography 212, A869
Gravimetric procedure 5
History of colorimetry 129
Infrared spectrometers (Chemical instrumentation) A5, A71
Inorganic infrared 201
Ionization constant of water. (TE) 260
Organic reagents for tin 410
Paper chromatography for ions 400
Pesticide residue control 370
Precipitation from homogeneous solution. 16
Salting-out chromatography 338
Saponification equivalents 611
Solubility and K_{sp} 460
Thermobalance 566, 568
Turbidimetry 358
Updating 455
Analytical chemistry and the non-analytical graduate student. William MacNevin 527
Analytical tool in organic chemistry, NMR spectroscopy as an. J. C. Martin 286
Antecedents to modern concepts of configurational symmetry in chemistry. Mel Gorman 99
Apparatus
Analog computer plugboard 492
Calorimetry 512
Cleaning burets 548
Constant current 519
Constant current supply 517
Conversion of hypodermics 626
Coulometer 361
DC source 521
Float method: dp/dc 578
For oxygen-sensitive solutions 264
Gas generator 19
Heating bath 102
Infrared spectrometers (Chemical instrumentation) A5, A71
Melting point 634
Micro-balance 469
Micro-tube filler 524
Plastic mounts for radioactive samples 208
Radioisotope filter 344
Rubber Stuart models 295
Safety device 525
Teflon stirrer bearing 577
Thermobalance 566, 568
Vacuum distillation 459
Variable transformer 96
Apparatus for oxygen-sensitive volumetric solutions. E. L. Martin and K. E. Bentley 264
Apparatus for the Friedel-Crafts reaction. Chester B. Kremer and Samuel H. Wilen 306
Approach to complex equilibrium problems. James N. Butler 141
Arsenate, Detection of phosphate in the presence of. Ramesh Chandra 408
Asking students to use facts. Donald C. Gregg 478
Aspirin, Analysis of: A conductometric titration. James S. Proctor and John E. Roberts 471
Association, Solvation and, from colligative properties by graphical means. George B. Smith, et al. 489
Atomic structure
2p orbitals 20
Atomic weights
^{12}C scale ..... 86, (E)131, (L)554
Table of 1961 values 625
Avogadros hypothesis
Anniversary 483
Avogadro's number ..... 51, 86, (E)131, 549, 551, (L)554, 555
Avogadro's number, estimation of molecular size and. "Visible" monomolecular films. Tested demonstration A751
Awards
Dexter address, 1960, Denis I. Duveen (P) 418
Scientific Apparatus Makers Association Award Address, 1961, John C. Bailar, Jr. (P, Cover) 434

Backgrounds of graduate school chemistry faculties. Raymond P. Mariella and Joan M. Cormack 427
Balance, micro, A rapid, direct-reading. Daniel L. Harris 469
Basis for a new conception of an electrode potential, The. J. B. Ramsey 353
Baxter, John F. (P) 369
Beginning chemistry course, Geometry in the. L. E. Strong, L. B. Clapp, and J. O. Edwards 530
Benzenoid nitro-compounds as organic reagents for tin(II). Robert L. Burns and Fred M. Gruen 410
Benzyne, The chemistry of. Joseph F. Bunnett 278
Berthollet, Claude Louis (P) 232
Bibliography, Personalized: An approach to the history of chemistry. Denis I. Duveen 418
Biochemistry
Electrophoresis experiment 36
Prebiological experiment 632
RNA and proteins 217
Vitalism history 614
Biochemistry laboratory experiment. Irwin Scher, Edward Walsh, and Barney Kadis 632
Biochemistry, Revised teaching of. Howard M. Myers 363
Biography
Boyle, Robert 106
Chichibabin, Aleksei E. 622
Dressel, Oskar 620
Fischer, Emil 93
Kekulé, August 118
Patterson, Robert Maskell 561
Personalized bibliography 418
Spring, Walther 422
Swarts, Frederic 423
Willstätter, Richard 10
Boiling liquid freezes water. Tested demonstration A813
Boltzman distribution law, A new derivation of. Reino W. Hakala 33
Bond energies, The uses and abuses of. Bruce E. Knox and Howard B. Palmer 292
Bonding
Models 606
2p orbitals 20
Bonding, chemical, Principles of. R. T. Sanderson 382
Bonding, Models illustrating types of orbitals and. Wilbur L. Baker 606
Books, Classified catalog of A646
Boyle, Robert (P) 106
Bromide, Chloride, and iodide determination. Marvin Wayne Hanson 412
Bucknell Conference, A report of: The undergraduate training of chemistry majors. Bennett R. Willeford, Jr., and Leallyn B. Clapp 251
Buffer series, An easily prepared wide range. Walter R. Carmody 559
Buildings, academic chemistry, New fundamental designs for. G. Ross Robertson 474
Burning phosphorus in air. Tested demonstration A23
Butanol-heptane mixtures, Viscosities of. K. Keith Innes 564

CACT—See *California Association of Chemistry Teachers*
Cadmium, Ion exchange separation of. Maurice M. Vick and E. Lyndol Harris 414
Cadmium, Non-cyanide detection of, in the presence of copper. Ramesh Chandra 409
Calculating molar solubilities from equilibrium constants. James N. Butler .. 460

California Association of Chemistry Teachers:
Articles
Antecedents to modern concepts of configurational symmetry in chemistry. MEL GORMAN ... 99
Gas Chromatography. ROBERT L. PECSOK ... 212
General philosophy of organic nomenclature. CHARLES D. HURD ... 43
New aspects of photosynthesis. J. A. BASSHAM ... 151
New fundamental designs for academic chemistry buildings. G. Ross ROBERTSON ... 474
Nuclear magnetic resonance spectroscopy. JOHN D. ROBERTS ... 581
Pesticide residue control under the Food, Drug, and Cosmetic Act. CURTIS R. JOINER ... 370
Role of chemistry in computers, The. A. H. SPORER ... 323
Summer conference, 1961 ... 266
Calorimeter, bomb, A flashbulb. FREDERICK M. HORNYAK ... 97
Calorimetry, precision solution, Temperature and power measurements in. WILLIAM F. O'HARA, ET AL. ... 512
Cannizzaro, Stanislao (P) ... 84
Karlsruhe Congress ... 83
Carbanions, stable, A convenient method for demonstration of. PETER T. LANSBURY ... 307
Carbon compounds, Stereoisomerism of. WILLIAM K. NOYCE ... 23
Carbon-14, half-life of, Redetermination of the. National Bureau of Standards Office of Technical Information ... 82
Carbonyl, metal, synthesis, Recent developments in. HAROLD E. PODALL ... 187
Catalysis
Metal oxides ... 312
Catalysis, Experiments in acid-base. J. A. LEISTEN ... 132
Catalytic oxidation of ammonia. Tested demonstration ... A23
Catalyzed reaction of phenyl isocyanate with butanol, The. A thermoanalytical kinetic experiment. L. B. WEISFELD ... 88
Chem Study—See Chemical education materials study
Chem gems ... 163, 398, 440, 491
Chemical calculations
Mole basis ... 551
Chemical education materials study, The. J. A. CAMPBELL ... 2
Chemical education—then and now. JOHN C. BAILAR, JR. ... 434
Chemical engineering program, A flexible undergraduate. JOHN J. MCKETTA AND ROBERT S. SCHECHTER ... 261
Chemical equilibria in imperfect gases. C. J. G. RAW ... 140
Chemical history
Latin America ... 628
Chemical information centers of Latin America. J. VIKIN ... 628
Chemical instrumentation. S. Z. LEWIN.
11. Infrared spectrometers ... A5, A71
12. Nuclear radiation detectors ... A135
13. Nuclear radiation electronic gear ... A225, A305, A389, A457
14. Chromatographic equipment ... A515, A567, A713, A789
15. Gas chromatographs ... A869
Chemical literature
Availability of nomenclature reports ... 301
Editorially speaking ... 223
Organic nomenclature ... 43
Punched cards ... 526
Russian abbreviations ... 41
Chemical projects: Research ideas for young chemists. JAY A. YOUNG AND JOHN K. TAYLOR.
Analytical ... A35A, A555, A612, A757
Biochemistry ... A611, A758, A820
Colloid ... A819, A891
Organic ... 9, A183, A421, A501, A556
Physical ... 8, A104, A263, A353, A502
Polymer ... A892
Qualitative ... A103, A184, A264, A422
Chemistry accidents in high school. ROBERT D. MACOMBER ... 367
Chemistry in computers, The role of. A. H. SPORER ... 323
Chemistry of benzyne, The. JOSEPH F. BUNNETT ... 278
Chemistry of creation of the heavy elements, The. CHARLES D. CORYELL ... 67
Chemistry of the solid state. Symposium. Introduction. ALLEN B. SCOTT ... 224
Chemistry program for teenagers, A summer. JOSEPH K. OBOLD AND THOMAS S. BRIGGS ... 47
Chemistry student and the chemical industry, The. JOSEPH S. SCHMUCKLER AND IRWIN SIEGELMAN ... 148
Chichibabin, Aleksei Evgen'evich. (P) E. CERKOVNIKOV ... 622
Chloride, bromide, and iodide determination. MARVIN WAYNE HANSON ... 412
Chlorine water substitute. Tested demonstration ... A813
Chromatograph, gas, for students, An inexpensive versatile multi-column. F. SICILIO, H. BULL, III, R. C. PALMER, AND J. A. KNIGHT ... 506
Chromatographic equipment. Chemical instrumentation. S. Z. LEWIN ... A515, A567, A713, A789
Chromatographic scheme, A paper, for the identification of metallic ions. A. S. RITCHIE ... 400
Chromatography, Gas. ROBERT L. PECSOK 212
Chromatography, paper, Ion exchange. ALAN T. THOMAS AND J. P. PHILLIPS ... 406
Chromatography, Salting-out: A review. WILLIAM RIEMAN III ... 338
Chromyl chloride test for chlorides, The. Textbook errors, 35. KAROL J. MYSELS ... 627
Chronology of nucleosynthesis and extinct natural radioactivity. TRUMAN P. KOHMAN ... 73
Cis-trans isomerization. Tested demonstration ... A179
Clapp, Leallyn B. (P) ... 637
Classified catalog of books ... A646
College chemistry course for non-science majors: Report of the Montana State Conference. A. B. GARRETT, W. B. COOK, AND W. T. LIPPINCOTT ... 253
Colligative properties, Solvation and association from, by graphical means. GEORGE B. SMITH, ET AL. ... 480
Colloid Chemistry
Adsorption ... 357
Rheology of macromolecules ... 110
Color change with temperature. Tested demonstration ... A23
Color filmstrips of atomic and molecular models (Review) ... 477
Colored slides, Dye solutions for the preparation of. LEONARD SPIALTER AND JONATHAN S. SMITH, II ... 473
Colorimetry, Development of the laws of. DOROTHY R. MALININ AND JOHN H. YOE ... 129
Combining volumes: Hydrogen chloride and ammonia. Tested demonstration. A813
Committee on status of the teaching of chemical literature, Report of (Division of Chemical Education, ACS) ... 273
Common ion effect on solubility, Radioisotope demonstration of. E. ROIG, ET AL. ... 350
Common ion effect. Tested demonstration ... A813
Complex formation, The equilibria of. Textbook errors, 32. JAMES E. BANKS 391
Complexes
Crystal field theory ... 164
Complexes, The structure and spectra of nickel(II) and copper(II). W. MANCH AND W. CONARD FERNELIUS ... 192
Computer instruction, Analog: A plugboard teaching aid. JAMES O. OSBURN ... 492
Computers, The role of chemistry in. A. H. SPORER ... 323
Concentration, H_2SO_4, Heat of reaction and. ENNO WOLTHUIS, ARIE LEEGWATER, AND JOHN VANDER PLOEG ... 472
Condensation of noxious gases. Tested demonstration ... A357
Condensed norms: ACS cooperative examinations. Division of Chemical Education ... 210, 211
Conductivity, Ultra low, water. National Bureau of Standards Summary Technical Report ... 421
Conductometric titration, A: Analysis of aspirin. JAMES S. PROCTOR AND JOHN E. ROBERTS ... 471
Configuration, electronic, of metal oxides. D. E. O'REILLY ... 312
Configurational symmetry in chemistry, Antecedents to modern concepts of. MEL GORMAN ... 99
Connecticut, Western, ACS Section, Educational activities of the. ALLAN E. SHERR ... 428
Constant current source for student use, A. CHARLES K. MANN AND VERNEL C. CHAMPEAUX ... 519
Constant current supply for electrokinetic experiments. STEPHEN W. TOBEY ... 517
Contemporary analysis and the chemistry curriculum. L. B. ROGERS ... 455
Content, Course, of general chemistry. HOWARD NECHAMKIN ... 255
Controlled atmosphere thermobalance. WESLEY W. WENDLANDT ... 566
Controller, Titration, for demonstrating the behavior of acid-base indicators. JOHN T. STOCK ... 633
Convenient method for demonstration of stable carbanions. PETER T. LANSBURY ... 307
Convenient radioactivity sources. D. J. CARSWELL ... 352
Convenient safety shield for small-scale apparatus. STANLEY H. LANGER AND PETER PANTAGES ... 634
Coordination chemistry in general chemistry texts. ROY W. CLARK AND JOEL SELBIN ... 466
Cope, Arthur C. (P) ... 589
Copper, A specific qualitative reagent for. T. R. WILLIAMS AND FREDERICK BURTON ... 413
Copper(II) complexes, The structure and spectra of nickel(II) and. W. MANCH AND W. CONARD FERNELIUS ... 192
Copper group, Rapid detection of cations of. RAMESH CHANDRA ... 413
Copper, Non-cyanide detection of cadmium in the presence of. RAMESH CHANDRA. 409
Corrosion, gaseous, of metals, Diffusion in solids and. WALTER J. MOORE ... 232
Cosmic abundances, Recent improvement in our knowledge of. WILLIAM D. EHMANN ... 53
Cosmochemistry. Symposium. Introduction. TRUMAN P. KOHMAN ... 52
Coulometer for titrations with externally generated reagents, An inexpensive. WILLIAM F. HEAD, JR. AND MAX M. MARSH ... 361
Course content of general chemistry, The. HOWARD NECHAMKIN ... 255
Creation of the heavy elements, The chemistry of. CHARLES D. CORYELL ... 67
Crookes tube, The paddle-wheel. Textbook errors, 33. J. ARTHUR CAMPBELL ... 480
Cryoscopy, Molecular weights by. ROBERT MIKULAK AND OLAF RUNQUIST ... 557
Crystal field theory—See Ligand field theory
Metal oxides ... 312
Crystal field theory and substitution reactions of metal ions. RALPH G. PEARSON ... 164
Crystal models, Paper-made. YASUYUKI KOMURO AND KŌZŌ SONE ... 580
Crystal structure
Order-disorder theory ... 538

Crystals, inorganic, The luminescence of. Ferd E. Williams..... 242
Crystals, Ionic. Howard W. Etzel..... 225
Current, constant, source for student use. Charles K. Mann and Vernel C. Champeaux..... 519
Current, constant supply for electrokinetic experiments. Stephen W. Tobey.... 517
Curriculum
Analytical for graduate students..... 527
Bucknell Conference report..... 251
Chemical engineering program..... 261
CHEMS program..... 2
Editorially speaking..... 223
Humanities and sciences..... 441
Modern analysis..... 455
Montana State Conference report..... 253
Radioisotopes..... 344
Cyclic, inorganic, compounds, A systematization of. I. Haiduc..... 134

Dalton's law of partial pressures. Tested demonstration..... A545
Decomposition, Pyrolytic, of carboxylate salts to ketones. Textbook Errors, 31. Harry P. Schultz and Jackson P. Sichels.....300, (L)638
Deionized water by electrodialysis. J. A. Schufle..... 17
Demonstrating enzymatic hydrolysis of glucosides by paper chromatography. S. F. Darling..... 508
Demonstration answer to a stereochemical puzzler. Tested demonstration..... A289
Demonstration of optical rotation with an "Eskimo Yo-Yo." S. J. Gill..... 263
Demonstration of stable carbanions, A convenient method for. Peter T. Lansbury..... 307
Demonstrations, filmed, Using in general chemistry. Ruth Botdorf..... 95
Depsides: A synthesis by Emil Fischer. C. V. Ratnam..... 93
Designs, New fundamental, for academic chemistry buildings. G. Ross Robertson..... 474
Detection of phosphate in the presence of arsenate. Ramesh Chandra..... 408
Determination of saponification equivalents of phenolic esters. S. W. Tobey, S. D. McGregor, and Sharon L. Cottrill. 611
Development of the laws of colorimetry. Dorothy R. Malinin and John H. Yoe..... 129
Device for charging micro-tubes with toxic reagents. John T. Stock, M. A. Fill and R. G. Bjork..... 524
Device to convert a hypodermic syringe into a multi-purpose apparatus. Samuel Shapiro..... 626
Diamond, The synthesis of. H. Tracy Hall..... 484
Differential thermal analysis, Reaction kinetics by. Wesley W. Wendlandt.. 571
Diffusion in solids and the gaseous corrosion of metals. Walter J. Moore..... 232
Diffusion under the microscope. Yasunori Nishijima and Gerald Oster..... 114
Dimethylmaleate, Synthesis of. Tested demonstration..... A179
Dipole moments of symmetrical organic molecules. Textbook errors, 34. Wilbur N. Moulton..... 522
Direct current source, low cost, for the laboratory. Robert Barnard and Ray Woodriff..... 521
Discovery of Germanin by Oskar Dressel and Richard Kothe, The. Jutta Dressel (Ralph E. Oesper, *Translator*)..... 620
Displacement reactions—spongy tin. Tested demonstration..... A637
Disposable plastic mounts for radioactive samples. T. E. Burgess, J. T. Looby, and W. C. Orr..... 208
Distillation, vacuum, Fractionation technique for. Jack Radell and J. W. Connolly..... 459

Division of Chemical Education of ACS
Committee on Institutes and Conferences, Report of..... 529
Committee on Status of Teaching of Chemical Literature, Report of..... 273
Condensed norms: ACS cooperative examinations..... 210
Message from the chairman. John F. Baxter..... 369
Nominations for office of chairman-elect, 1961–62..... 273
Officers for 1961–62..... 437
Treasurer's report. William G. Kessel. 635
Dressel, Oskar, and Kothe, Richard (P)..... 620
Dressel, Oskar, and Richard Kothe, The discovery of Germanin by. Jutta Dressel (Ralph E. Oesper, *Translator*)..... 620
Dye s[illegible]tions for the preparation of colored slides. Leonard Spialter and Jonathan S. Smith II..... 473

Easily prepared wide range buffer series. Walter R. Carmody..... 559
Editorially speaking.....1, 51, 105, 159, 223, 277, 333, 381, 433, 483, 537, 589
Education, Chemical—then and now. John C. Bailar, Jr..... 434
Educational activities of the Western Connecticut ACS Section. Allan E. Sherr..... 428
Effect of temperature on solubility. Tested demonstration..... A99
Electrochemistry
Conductances in amines..... 590
Deionized water..... 17
Electrode potential..... 353
Fuel cells..... 267
Potentials and demonstrations..... A637
Three-dimensional representation..... 149
Transference apparatus..... 516
Electrochemistry of the solid state. Allen B. Scott..... 317
Electrode potential, The basis for a new conception of an. J. B. Ramsey..... 353
Electrode potentials with a vacuum tube voltmeter. Tested demonstration.... A637
Electrodeposition, Three-dimensional representation of. W. H. Reinmuth..... 149
Electrodialysis, Deionized water by. J. A. Schufle..... 17
Electrokinetic experiments, Constant current supply for. Stephen W. Tobey.. 517
Electron paramagnetic resonance of free radicals. Juan A. McMillan..... 438
Electronic configuration of metal oxides. D. E. O'Reilly..... 312
Electrons
Editorially speaking..... 105
Electrons in Atoms, There are! Anatol Sementsov..... 22
Electrophoresis
Equipment survey.....A713, A789
Electrophoresis, filter paper, Student experiment with. James E. Garvin..... 36
Elements, heavy, The chemistry of creation of the. Charles D. Coryell..... 67
Elements of order-disorder theory: Applications to gas adsorption, binary solutions, alloys, and ferromagnetism. J. M. Honig..... 538
Empirical formula (L). P. M. Laughton.. 378
Endothermic adsorption, The existence of. Textbook errors, 29. John M. Thomas 138
Energies, bond, The uses and abuses of. Bruce E. Knox and Howard B. Palmer..... 292
Entropy
Will a reaction occur?..... 334
Enzymatic hydrolysis of glucosides by paper chromatography, Demonstrating. S. F. Darling..... 508
Equilibria, Chemical, in imperfect gases. C. J. G. Raw..... 140
Equilibria of complex formation, The. Textbook errors, 32. James E. Banks. 391

Equilibrium constants, Calculating molar solubilities from. James N. Butler... 460
Equilibrium problems, complex, An approach to. James N. Butler..... 141
Equivalent weight
Definition.....(L)637
Equivalents, Moles and: Quantities of matter. Irwin Cohen..... 555
Errors, Ratio, in pseudo first order reactions. Fred Sicilio and M. D. Peterson.... 576
"Eskimo yo-yo," A demonstration of optical rotation with an. S. J. Gill..... 263
Examinations
Quantitative practical..... 144
Examinations, ACS cooperative, Condensed norms..... 211
Existence of endothermic adsorption. Textbook errors, 29. John M. Thomas.... 138
Experiments in acid-base catalysis. J. A. Leisten..... 132
Extensive properties, A geometric approach to. S. C. Nyburg and H. F. Halliwell.....123, (L)329, (E)329

Faculties, graduate school chemistry, Backgrounds of. Raymond P. Mariella and Joan M. Cormack..... 427
Faraday's laws..... 552
Faraday's laws in one equation. F. C. Strong..... 98
Ferric ion, Graphical representation of hydrolysis of the. G. F. Atkinson and W. A. E. McBryde..... 127
Ferromagnetism, gas adsorption, binary solutions, and alloys, Applications to. Elements of order-disorder theory. J. M. Honig..... 538
Filmed demonstrations in general chemistry, Using. Ruth Botdorf..... 95
Films, "Visible" monomolecular: Estimation of molecular size and Avogadro's number. Tested demonstration A751
Filter paper electrophoresis, Student experiment with. James E. Garvin..... 36
Filtering, Rapid, without using a filter pump. Tested demonstration..... A813
Filtration technique, Simple, for gravimetric determinations. Shirley W. Gaddis. 5
Fire under water. Tested demonstration.. A751
Flashbulb bomb calorimeter. Frederick M. Hornyak..... 97
Flexible undergraduate chemical engineering program. John J. McKetta and Robert S. Schechter..... 261
Float method for the analysis of solutions. James O. Osburn and Il C. Choi.... 578
Fluidized solids, Laboratory heating bath using. Milton Manes and Davis G. Chalmers..... 102
Food, Drug, and Cosmetic Act, Pesticide residue control under the. Curtis R. Joiner..... 370
Fractionation technique for vacuum distillation. Jack Radell and J. W. Connolly..... 459
Franklin Institute chemistry exhibit: A design for learning. Irwin Siegelman and Joseph S. Schmuckler..... 597
Frederic Swarts (1866–1940). (P) Jean Timmermans (Ralph E. Oesper, *Translator*) 423
Free radicals, Electron paramagnetic resonance of. Juan A. McMillan..... 438
Frequency shifts in strained ring double bonds. Vibrating molecular models. Norman B. Colthup..... 394
Friedel-Crafts reaction, Apparatus for the. Chester B. Kremer and Samuel H. Wilen..... 306
Fuel cells—electrochemical converters of chemical to electrical energy. Joseph Weissbart..... 267
Fumes, nitric acid, A simple test for. Alfred Chan, Jr..... 412

Gas chromatograph for students, An inexpensive versatile multi-column. F. Sicilio, H. Bull, III, R. C. Palmer, and J. A. Knight..... 506

Gas chromatographs. Chemical instrumentation. S. Z. Lewin A869
Gas chromatography. Robert L. Pecsok. 212
Gas generator for elementary chemistry, An improved. G. W. Thiessen and M. Hall 19
Gases, noxious, Condensation of. Tested demonstration A357
General Chemistry
Bonding principles 382
Cryoscopy experiment 557
Electrons in atoms 22
Elemental qualitative analysis 607
Entropy (editorial) 333
Entropy 334
Faraday's laws 98
Geometry 530
Hydrogen from Al-Zn alloy 558
Mole ..... 51, 86, (E)131, 549, 551, (L)554, 555
Redox survey ..... 326, (L)637
Using filmed demonstrations 95
General chemistry, course content of. Howard Nechamkin 255
General Chemistry, Form 1960. Reviews of ACS Cooperative Exams. S. L. Burson, Jr. 210
General chemistry texts, Coordination chemistry in. Roy W. Clark and Joel Selbin 466
General Electric Science Fellowship Program at Union College: A pioneer summer institute. E. K. Bacon 39
General philosophy of organic nomenclature. Charles D. Hurd 43
Geochemistry
Cosmochemistry symposium 52
Geometric approach to extensive properties, A. S. C. Nyburg and H. F. Halliwell ..... (L)329, (E)329
Geometric approach to the Gibbs-Duhem equation, A. Lawrence F. Beste 509
Geometry in the beginning chemistry course. L. E. Strong, L. B. Clapp, and J. O. Edwards 530
Germanin, The discovery of, by Oskar Dressel and Richard Kothe. Jutta Dressel (Ralph E. Oesper, *translator*) 620
Ghent, Kekulé's life at (1858–1867). J. Gillis. (Ralph E. Oesper, *translator*) 118
Gibbs-Duhem equation, A geometric approach to the. Lawrence F. Beste 509
Glaser, Carl (P) 120
Glucosides by paper chromatography, Demonstrating enzymatic hydrolysis of. S. F. Darling 508
Graduate school chemistry faculties, Backgrounds of. Raymond P. Mariella and Joan M. Cormack 427
Graduate student, Analytical chemistry and the non-analytical. William MacNevin 527
Graham's law of diffusion using overhead projector. Tested demonstration A23
Graphical means, Solvation and association from colligative properties by. George B. Smith, et al. 489
Graphical representation of hydrolysis of the ferric ion. G. F. Atkinson and W. A. E. McBryde 127
Gravimetric determinations, A simple filtration technique for. Shirley W. Gaddis 5
Group experiment on the Hammett sigma-rho relation. J. A. Leisten 302

Half life of UX_1(Pa-234), Measurement of, with a dipping counter. Jerry Braunstein and Raymond H. Young. 31
Hammett sigma-rho relation, A group experiment on the. J. A. Leisten 302
Heat of precipitation: A general chemistry experiment. H. Lawrence Clever .. 470
Heat of reaction and H_2SO_4 concentration: A general chemistry experiment. Enno Wolthuis, Arie Leegwater, and John Vander Ploeg 472
Heating bath, Laboratory, using fluidized solids. Milton Manes and David G. Chalmers 102
Henry, L.
Controversy with Markovnikov 297
High School chemistry
Accidents 367
CHEMS program 2
Modified qualitative analysis 146
Projects (Editorial) 159, 277
Pupils in industry 148
High school students, A summer research participation program for. Reed F. Riley and C. G. Overberger 424
History of Chemistry
Avogadro sesquicentennial (Editorially speaking) 483
Chichibabin, Aleksei E. 622
Colorimetry laws 129
Configurational symmetry 99
Crookes tube 480
Discovery of Germanin 620
Entropy concept 334
Karlsruhe Congress 83
Kekulé at Ghent 118
Liebig-Pasteur 614
Markovnikov rule 297
Patterson, Robert Maskell 561
Personalized bibliography 418
"The Sceptical Chymist" 106
Spring, Walther 422
Swarts, Frederic 423
Willstätter, Richard 10
Homogeneous solution, Precipitation from: A lecture demonstration. Louis Gordon and Eugene D. Salesin 16
How can you tell whether a reaction will occur? George E. MacWood and Frank H. Verhoek 334
Humanities, The problem of the sciences and the. Harold G. Cassidy 441
Hydride formation by atomic hydrogen reactions. Bernard Siegel 496
Hydrolysis of the ferric ion, Graphical representation of. G. F. Atkinson and W. A. E. McBryde 127
Hypodermic syringe into a multi-purpose apparatus, Device to convert a. Samuel Shapiro 626

Identification analysis, Solid sample, in high school chemistry. William Vasilakes 146
Identification of metallic ions, A paper chromatographic scheme for the. A. S. Ritchie 400
Imperfect gases, Chemical equilibria in. C. J. G. Raw 140
Improved gas generator for elementary chemistry. G. W. Thiessen and M. Hall 19
Incorporating radioisotope techniques into the chemistry curriculum. Norman S. Radin 344
Industrial chemistry
Deionized water 17
Inexpensive coulometer for titrations with externally generated reagents. William F. Head, Jr., and Max M. Marsh 361
Inexpensive Stuart-type molecular models. William G. Hoover and Duward Schriver 295
Inexpensive versatile multi-column gas chromatograph for students. F. Sicilio, H. Bull, III, R. C. Palmer, and J. A. Knight 506
Infrared spectrometers. Chemical instrumentation. S. Z. Lewin A5, A71
Infrared spectroscopy, Inorganic. John R. Ferraro 201
Inorganic chemistry
Analysis by synthesis 407
Bonding 382
Copper complexes ..... (L)638
Diamond synthesis 484
Hydride formation 496
Introductory geometry 530
Iron(III) hydrolysis 127
Lithium by electrolysis (TD) A419
Metal-in-amine solutions 590
Metal oxides 312
Non-stoichiometric compounds 90
Oxide reduction 117
Semiconductors 238
Silver complex electrolysis (TD) A419
Solid state symposium 224
Solubility and K_{sp} 460
Text treatment of coordination 466
Inorganic chemistry, Recent advances in. Symposium 160
Inorganic crystals, The luminescence of. Ferd E. Williams 242
Inorganic cyclic compounds, A systematization of. I. Haiduc 134
Inorganic infrared spectroscopy. John R. Ferraro 201
Inorganic materials, solid state, Recent advances in. Otto L. Forchheimer and Terry F. Newkirk 183
Institutes and conferences, Report of the committee on. Division of Chemical Education, ACS. W. T. Lippincott, *Chairman* 529
Institutes in the academic year: Review and future plans. Lewis N. Pino and Robbin C. Anderson 451
Institutes, Summer: Review and future plans. William E. Morrell 448
Institutional influences in the graduate training of eminent chemists. B. R. Siebring 630
Instruction, Programmed, in chemistry. Jay A. Young 463
Intermediates, The reactive, of organic chemistry. Ross Stewart 308
Introduction to Le Chatelier's principle. Tested demonstration A491
Iodide, Chloride, and bromide determination. Marvin Wayne Hanson 412
Ion exchange paper chromatography. Alan T. Thomas and J. P. Phillips 406
Ion exchange-radiochemical experiment. J. F. Mucci, Dorothy E. Spiegel, and Robert L. Stearns 348
Ion exchange separation of cadmium. Maurice M. Vick and E. Lyndol Harris 414
Ionic crystals. Howard W. Etzel 225
Ionization constant of water. Textbook errors, 30. Thedford P. Dirkse 260
Iron, Transition temperature in. Tested demonstration A357
Is there an alternative to pH? Francis E. Crane, Jr. ..... 365, (L)638
Isomerization, Cis-trans. Tested demonstration A179
Isopiestic vapor pressure apparatus. G. W. Dailey, R. B. Huff, J. Kang, L. D. Queen and C. S. Patterson 28
Isotopes
Cosmic abundance 53
IUPAC reports on symbolism and nomenclature 301
Report on atomic weights 625

Karlsruhe Congress: A centennial retrospect. Aaron J. Ihde 83
Kekulé, August ..... 83, (P)120
Kekulé's life at Ghent (1858–1867). J. Gillis. (Ralph E. Oesper, *translator*) 118
Kessel, William G. (P) 635
Ketones, The pyrolytic decomposition of carboxylate salts to. Textbook errors, 31. Harry P. Schultz and Jackson P. Sichels 300
Kinetic experiment, A thermoanalytical: The catalyzed reaction of phenyl isocyanate with butanol. L. B. Weisfeld 88
Kinetics
Diamond synthesis 484
Hammett equation experiment 302
Kinetics, Nomographs for first and second order. S. G. Smith and I. D. R. Stevens 574

Kinetics, Reaction, by differential thermal analysis. Wesley W. Wendlandt... 571
Körner, Wilhelm (P)..... 120
Kothe, Richard, and Dressel, Oskar (P)..... 620
Kothe, Richard, The discovery of Germanin by Oskar Dressel and. Jutta Dressel (Ralph E. Oesper, *Translator*)..... 620

Laboratory apparatus
Friedel-Crafts reaction..... 306
Hospital castoffs..... 619
Radioactivity sources..... 352
Safety shield..... 634
Titration demonstrations..... 633
Laboratory experiments
Acid-base catalysis..... 132
Adsorption isotherm..... 357
Alloy analysis..... 558
Aspirin by conductivity..... 471
Buffer series..... 559
Common ion-radiochemistry..... 350
Cryoscopy..... 557
Deamidation of Vitamin B_{12}..... 37
Diffusion under the microscope..... 114
Dyes..... 473
Enzymatic hydrolysis..... 508
Filtration technique..... 5
Flashbulb calorimeter..... 97
Hammett sigma-rho..... 302
Heat of precipitation..... 470
Heat of reaction..... 472
Ion exchange-radiochemistry..... 348
Isopiestic vapor pressure apparatus..... 28
Kinetics by DTA..... 571
Paper electrophoresis..... 36
Polystyrene synthesis..... 304
Prebiological chemistry..... 632
Precipitation and solubility..... 397
Thermoanalytical kinetic..... 88
Transference by moving boundary..... 516
Turbidimetry..... 358
Viscosities of mixtures..... 564
Laboratory heating bath using fluidized solids. Milton Manes and David G. Chalmers..... 102
Laboratory practical examination in quantitative chemistry. William M. MacNevin..... 144
Langmuir adsorption isotherm, Surface area of activated charcoal by. Boleslaw Ludwik Dunicz..... 357
Latin America, Chemical information centers of. J. Vikin..... 628
Le Chatelier's principle in chemical reactions, A systematic way to avoid. Lewis Katz..... 375
Le Chatelier's principle, Introduction to. Tested demonstration..... A491
Lead, The turbidimetric determination of. Gerald Perkins, Jr., et al..... 358
Lecture demonstrations
Optical rotation..... 263
Precipitation and solubility..... 397
Precipitation from homogeneous solution. 16
Stable carbanions..... 307
Lewis acid-base chemistry, A reaction to introduce. Tested demonstration.... A491
Liebig birthplace memorial. Ralph E. Oesper..... 122
Liebig-Pasteur controversy, The: Vitality without vitalism. George E. Hein.. 614
Ligand field theory—See crystal field theory
Structure and spectra..... 192
Lithium, preparation of metallic. Tested demonstration..... A419
Little-languages of science, Translating and re-jargonizing the. Jack De Ment... 415
Low cost direct current source for the laboratory. Robert Barnard and Ray Woodriff..... 521
Luminescence of inorganic crystals, The. Ferd E. Williams..... 242

Machine-sorted punched card systems, Adapting to hand sorting and coding. Stanley Kirschner..... 526
Majors, Chemistry, The undergraduate training of. Bennett R. Willeford, Jr., and Leallyn B. Clapp..... 251
Markovnikov rule, The. Gurnos Jones... 297
Mayer, Auguste (P)..... 120
Measurement of the half life of UX_2(Pa-234) with a dipping counter. Jerry Braunstein and Raymond H. Young 31
Mechanisms, Oxidation reduction. Frederick R. Duke..... 161
Melting point apparatus. Carlton S. Graves..... 634
Metal carbonyl synthesis, Recent developments in. Harold E. Podall..... 187
Metal-in-amine solutions, The nature of. E. Charles Evers..... 590
Metal oxides, Electronic configuration of. D. E. O'Reilly..... 312
Metallic ions, A paper chromatographic scheme for the identifications of. A. S. Ritchie..... 400
Metals, Diffusion in solids and the gaseous corrosion of. Walter J. Moore..... 232
Meteorites, origin of, Theories on the. Edward Anders and Gordon G. Goles..... 58
Micro-balance, A rapid, direct-reading. Daniel L. Harris..... 469
Microscope, Diffusion under the. Yasunori Nishijima and Gerald Oster... 114
Micro-tubes, Device for charging with toxic reagents. John T. Stock, et al. 524
Miscellanea No. 4. Textbook errors, 35. Karol J. Mysels..... 627
Mixtures, butanol-heptane, Viscosities of. K. Keith Innes..... 564
Mnemonics, More. Benjamin Becker.... 42
Model for optical rotation. Leon L. Jones and Henry Eyring..... 601
Models
Cast rubber..... 295
Molecular orbitals..... 502
Tetrahedral angle layout (L)..... 533
Models, crystal, Paper-made. Yasutuki Komuro and Kôzô Sone..... 580
Models illustrating types of π-orbitals and bonding. Wilbur L. Baker..... 606
Models, Vibrating molecular: Frequency shifts in strained ring double bonds. Norman B. Colthup..... 394
Mole
Editorially speaking..... 51
Collected comments..... 549
"Mole," A redefinition of. Shiu Lee..... 549
Mole and related quantities, The. E. A. Guggenheim.....86, (E)131, (L)554
Mole in quantitative chemistry, The. George Novello Copley..... 551
Molecular models featuring molecular orbitals. George C. Brumlik..... 502
Molecular models, Inexpensive Stuart-type. William G. Hoover and Duward Shriver..... 295
Molecular models, Vibrating: Frequency shifts in strained ring double bonds. Norman B. Colthup..... 394
Molecular oribitals, Molecular models featuring. George C. Brumlik..... 502
Molecular structure
Bonding..... 382
Molecular weights by cryoscopy. Robert Mikulak and Olaf Runquist..... 557
Moles and equivalents: Quantities of matter. Irwin Cohen..... 555
Monomolecular films, "Visible": Estimation of molecular size and Avogadro's number. Tested demonstration..... A751
"Monovalent oxygen" compounds. Textbook errors, 35. Karol J. Mysels... 627
Montana State Conference, A report of the: The college chemistry course for non-science majors. A. B. Garrett, et al..... 253
Motor-driven variable transformer, A. Meyer M. Markowitz, Daniel A. Bortta, and George Capriola..... 96
Museums
Franklin Institute..... 597

National Science Fair-International and the ACS Chemistry Awards. Robert M. Brooker and Richard T. Rapala. 209
National Science Foundation
Academic year institutes..... 451
Summer institutes..... 448
Nature of metal-in-amine solutions, The. E. Charles Evers..... 590
NEACT—See New England Association of Chemistry Teachers
New aspects of photosynthesis. J. A. Bassham..... 151
New derivation of the Boltzman distribution law. Reino W. Hakala..... 33
New England Association of Chemistry Teachers:
Articles
Asking students to use facts. Donald C. Gregg..... 478
Fuel cells—electrochemical converters of chemical to electrical energy. Joseph Weissbart..... 267
Geometry in the beginning chemistry course. L. E. Strong, L. B. Clapp, and J. O. Edwards..... 530
Redox revisited. *Karl L. Lockwood*... 326
Ribonucleic acid and protein synthesis. Jay S. Roth..... 217
Systematic way to avoid Le Chatelier's principle in chemical reactions. Lewis Katz..... 375
Officers for 1961–62..... 378
Official business..... 636
Twenty-third summer conference.. 272, (P)636
New fundamental designs for academic chemistry buildings. G. Ross Robertson..... 474
New qualitative reagent for aluminum, A. Carl T. Jones and Max B. Williams 411
New selective reducing agents. Herbert C. Brown..... 173
Nickel(II) and copper(II) complexes, The structure and spectra of. W. Manch and W. Conard Fernelius..... 192
Nitric acid fumes, A simple test for. Alfred Chan, Jr..... 412
Nitrogen-fluorine chemistry, Recent developments in. Charles B. Colburn 180
NMR spectroscopy as an analytical tool in organic chemistry. J. C. Martin.... 286
Nomenclature, organic, The general philosophy of. Charles D. Hurd..... 43
Nominations for office of chairman-elect, 1961–62. Division of Chemical Education, ACS..... 273
Nomographs for first and second order kinetics. S. G. Smith and I. D. R. Stevens 574
Non-cyanide detection of cadmium in the presence of copper. Ramesh Chandra .....409, (L)638
Non-metallic compounds, Non-stoichiometry in. Leo Brewer..... 90
Non-science majors, The college chemistry course for. A. B. Garrett, W. B. Cook, and W. T. Lippincott..... 253
Non-stoichiometry in non-metallic compounds. Leo Brewer..... 90
Notes on qualitative analysis. A Chem Ed Collection..... 406
Nuclear chemistry
Nucleogenesis..... 67
Nuclear magnetic resonance spectroscopy. John D. Roberts..... 581
Nuclear radiation detectors. Chemical instrumentation. S. Z. Lewin..... A135
Nuclear radiation electronic gear. Chemical instrumentation. S. Z. Lewin.... .....A225, A305, A389, A457
Nucleosynthesis and extinct natural radioactivity, Chronology of. Truman P. Kohman..... 73

Optical rotation, A model for. Leon L. Jones and Henry Eyring..... 601
Optical rotation with an "Eskimo yo-yo," A demonstration of. S. J. Gill..... 263

Orbitals and bonding, Models illustrating types of. WILBUR L. BAKER..... 606
Orbitals, molecular, Molecular models featuring. GEORGE C. BRUMLIK..... 502
Orbitals, The shape of the 2p and related. Textbook errors, 28. IRWIN COHEN.. 20
Order-disorder theory, Elements of: Applications to gas adsorption, binary solutions, alloys, and ferromagnetism. J. M. HONIG..... 538
Organic chemistry
Acid-base catalysis experiments..... 132
Analysis for phosphorus..... 414
Benzyne..... 278
Bond energies..... 292
Carbanions demonstration..... 307
Chichibabin, Aleksei E..... 622
Cis-trans isomerization. Tested demonstration..... A179
Cyclopentanone synthesis..... (L)638
Demonstration of optical rotation..... 263
Depsides..... 93
Dipole moments (TE)..... 522
Enzymatic hydrolysis..... 508
Friedel-Crafts apparatus..... 306
Germanin discovery..... 620
Hammett equation experiment..... 302
History of Markovnikov rule..... 297
NMR spectroscopy..... 581
NMR use..... 286
Pesticides..... 370
Photosynthesis..... 151
Polystyrene synthesis experiment..... 304
Pyrolysis of carboxylate salts. (TE) ..... 300, (L)638
Reactive intermediates..... 308
Salting-out chromatography..... 338
Saponification equivalents..... 611
Selective reducing agents..... 173
Stereoisomerism..... 23
Synthesis of dimethylmaleate. Tested demonstration..... A179
Vitalism history..... 614
Organic compounds, A simple test for the detection of phosphorus in. ROGER KETCHAM AND ANNE DE G. LOW-BEER. 414
Organic nomenclature, The general philosophy of. CHARLES D. HURD..... 43
Organic reagents for tin(II), Benzenoid nitrocompounds as. ROBERT L. BURNS AND FRED M. GRUEN..... 410
Origin of meteorites, Theories on the. EDWARD ANDERS AND GORDON G. GOLES..... 58
Overhead projector, Graham's law of diffusion using. Tested demonstration.... A23
Oxidation-reduction mechanisms. FREDERICK R. DUKE..... 161
Oxygen-sensitive volumetric solutions, Apparatus for. E. L. MARTIN AND K. E. BENTLEY..... 264

PADDLE-wheel Crookes tube, The. Textbook errors, 33. J. ARTHUR CAMPBELL 480
Paper chromatographic scheme for the identification of metallic ions, A. A. S. RITCHIE..... 400
Paper chromatography, Demonstrating enzymatic hydrolysis of glucosides by. S. F. DARLING..... 508
Paper chromatography, Ion exchange. ALAN T. THOMAS AND J. P. PHILLIPS... 406
Paper electrophoretic study of rates of consecutive reactions: The deamidation of vitamin B_{12}. GRANT H. BARLOW..... 37
Paper-made crystal models. YASUYUKI KOMURO AND KŌZŌ SONE..... 580
Paramagnetic, Electron, resonance of free radicals. JUAN A. MCMILLAN..... 438
Partial pressures, Dalton's law of. Tested demonstration..... A545
Pasteur-Liebig controversy: Vitality without vitalism. GEORGE E. HEIN..... 614
Patterson, Robert Maskell: First to teach atomic theory in America? (P) WYNDHAM D. MILES..... 561
Periodic table, A qualitative elemental analysis system correlated with the. ERNEST H. SWIFT AND WILLIAM P. SCHAEFER..... 607
Periodic table display..... 322
Personalized bibliography: An approach to the history of chemistry. DENIS I. DUVEEN..... 418
Pesticide residue control under the Food, Drug, and Cosmetic Act. CURTIS R. JOINER..... 370
pH, Is there an alternative to? FRANCIS E. CRANE, JR.....365, (L)638
Philosophy of science
Humanities and sciences..... 441
Philosophy, The general, of organic nomenclature. CHARLES D. HURD..... 43
Phosphate, Detection of, in the presence of arsenate. RAMESH CHANDRA..... 408
Phosphorus in air, Burning. Tested demonstration..... A23
Phosphorus in organic compounds, A simple test for the detection of. ROGER KETCHAM AND ANNE DE G. LOW-BEER. 414
Photochemical catalysis of an aliphatic substitution reaction. Tested demonstration..... A289
Photosynthesis, New aspects of. J. A. BASSHAM..... 151
Physical Chemistry
Boltzman distribution law..... 33
Bond energies..... 292
Diamond synthesis..... 484
Dipole moments (TE)..... 522
Electrochemistry of the solid state..... 317
Electrode potential..... 353
Electron paramagnetic resonance review. 438
Endothermic adsorption..... 138
Errors in kinetics..... 576
Extensive properties.....123, (L)329, (E)329
Finding slopes..... 570
Fuel cells..... 267
Gas dynamics..... 257
Ionization constant of water. (TE).... 260
Kinetics by DTA experiment..... 571
Kinetics experiment.....88, 302
Kinetics nomographs..... 574
Metal oxide theory..... 312
Molecular vibrations..... 394
NMR spectroscopy..... 581
Optical rotations..... 601
Order-disorder theory..... 538
Photosynthesis..... 151
Solid state symposium..... 224
Solvation and association..... 489
Transference experiment..... 516
Viscosity of mixtures..... 564
Pioneer summer institute: The G.E. Science Fellowship Program at Union College. E. K. BACON..... 39
Plastic mounts for radioactive samples, Disposable. T. E. BURGESS, ET AL... 208
Plating from an iodide complex, Silver. Tested demonstration A419
Plugboard teaching aid: Analog computer instruction. JAMES O. OSBURN..... 492
Polystyrene—a multistep synthesis. SAMUEL H. WILEN, CHESTER B. KREMER, AND IRVING WALTCHER..... 304
Potentials, Electrode, with a vacuum tube voltmeter. Tested demonstration.... A637
Practical examination, laboratory, in quantitative chemistry. WILLIAM M. MACNEVIN..... 144
Precipitation and solubility effects, A simple demonstration of some. E. MATIJEVIC, J. P. KRATOHVIL, AND M. KERKER..... 397
Precipitation from homogeneous solution: A lecture demonstration. LOUIS GORDON AND EUGENE D. SALESIN.... 16
Precipitation, Heat of: A general chemistry experiment. H. LAWRENCE CLEVER.. 470
Preparation of metallic lithium. Tested demonstration..... A419
Preparative separations replace typical qualitative analysis. CARL MAHR.... 407
Principles of chemical bonding. R. T. SANDERSON..... 382
Problem of the sciences and the humanities, The. HAROLD G. CASSIDY..... 441
Programmed instruction in chemistry: An invitation to participate. JAY A. YOUNG..... 463
Projects, Chemical—See *Chemical projects*
Protein synthesis, Ribonucleic acid and. JAY S. ROTH..... 217
Pseudo first order reactions, Ratio errors in. FRED SICILIO AND M. D. PETERSON..... 576
Pumps, vacuum diffusion, Safety device for. G. O. PRITCHARD AND G. H. MILLER.. 525
Punched card systems, machine-sorted, Adapting to hand sorting and coding. STANLEY KIRSCHNER..... 526
Pyrolytic decomposition of carboxylate salts to ketones, The. Textbook errors. 31. HARRY P. SCHULTE AND JACKSON P. SICHELS.....300, (L)638

Qualitative analysis
Chromatographic scheme..... 400
Experimental method.....(L)102
K_{sp} variation..... 397
Sample identification..... 146
Qualitative analysis, Form 1960. Reviews of ACS Cooperative Exams. RICHARD E. FRANK..... 210
Qualitative analysis, Notes on. A Chem Ed Collection..... 406
Qualitative analysis, typical, Preparative separations replace. CARL MAHR.... 407
Qualitative demonstration of Raoult's law. Tested demonstration..... A545
Qualitative elemental analysis system correlated with the periodic table. ERNEST H. SWIFT AND WILLIAM P. SCHAEFER... 607
Quantitative chemistry. A laboratory practical examination in. WILLIAM M. MACNEVIN..... 144
Quantitative chemistry, The mole in. GEORGE NOVELLO COPLEY..... 551
Quantum mechanics
Optical rotations..... 601

RADIATION detectors, Nuclear. Chemical instrumentation. S. Z. LEWIN..... A135
Radiation, Nuclear, electronic gear. Chemical instrumentation. S. Z. LEWIN... .....A225, A305, A389, A457
Radioactive samples, Disposable plastic mounts for. T. E. BURGESS, ET AL.... 208
Radioactivity, extinct natural, Chronology of nucleosynthesis and. TRUMAN P. KOHMAN..... 73
Radioactivity sources, Convenient. D. J. CARSWELL..... 352
Radiochemical experiment, Ion exchange. J. F. MUCCI, ET AL..... 348
Radiochemistry
Chemical instrumentation..... A225
Half life of UX_2(Pa-234)..... 31
Resolving time.....29, (L)329
Techniques..... 344
Radioisotope demonstration of common ion effect on solubility. E. ROIG, I. G. RIECKEHOFF, C. S. RUSSO, AND J. D. CURET..... 350
Radioisotope techniques into the chemistry curriculum, Incorporating. NORMAN S. RADIN..... 344
Raoult's law, A qualitative demonstration of. Tested demonstration..... A545
Rapid detection of cations of copper group. RAMESH CHANDRA..... 413
Rapid, direct-reading micro-balance, A. DANIEL L. HARRIS..... 469
Rapid efficient method for reducing certain metallic isotopic oxides. PHILIP S. BAKER..... 117
Rapid filtering without using a filter pump. Tested demonstration..... A813
Rates of consecutive reactions, Paper electrophoretic study of: The deamidation of vitamin B_{12}. GRANT H. BARLOW.. 37

Ratio errors in pseudo first order reactions. Fred Sicilio and M. D. Peterson... 576
Reaction kinetics by differential thermal analysis. Wesley W. Wendlandt... 571
Reaction to introduce Lewis acid-base chemistry. Tested demonstration..... A491
Reactive intermediates of organic chemistry. Rose Stewart..... 308
Recent advances in inorganic chemistry. Symposium. Introduction. R. W. Parry..... 160
Recent advances in solid state inorganic materials. Otto L. Forchheimer and Terry F. Newkirk..... 183
Recent developments in metal carbonyl synthesis. Harold E. Podall..... 187
Recent developments in N—F chemistry. Charles B. Colburn..... 180
Recent developments in semiconductors. W. Crawford Dunlap..... 238
Recent improvements in our knowledge of cosmic abundances. William D. Ehmann..... 53
Redefinition of "Mole," A. Shiu Lee..... 549
Redetermination of the half-life of carbon-14. National Bureau of Standards Office of Technical Information..... 82
Redox revisited. Karl L. Lockwood .....326, (L)637
Reducing agents, New selective. Herbert C. Brown..... 173
Reducing certain metallic isotopic oxides, A rapid efficient method for. Philip S. Baker..... 117
Re-jargonizing the little-languages of science, Translating and. Jack De Ment..... 415
Research ideas for young chemists—*See Chemical projects*
Research participation program for high school students, A summer. Reed F. Riley and C. G. Overberger..... 424
Residue, Pesticide, control under the Food, Drug, and Cosmetic Act. Curtis R. Joiner..... 370
Resolving time in G-M counting, Using the decay of Ba-137 to determine. Robert L. Stearns and Joseph F. Mucci.....29, (L)329
Reviews of ACS Cooperative Exams..... 210
Revised teaching of biochemistry. Howard M. Myers..... 363
Rheology in the world of neglected dimensions. John D. Ferry..... 110
Ribonucleic acid and protein synthesis. Jay S. Roth..... 217
Richard Willstätter. Rolf Huisgen..... 10
Robert Maskell Patterson: First to teach atomic theory in America? Wyndham D. Miles..... 561
Role of chemistry in computers, The. A. H. Sporer..... 323
Rotation, optical, A model for. Leon L. Jones and Henry Eyring..... 601
Russian chemical literature, Abbreviations in. Julian F. Smith..... 41

Safety
Reaction shield..... 634
Test for acid wash..... 291
Safety device for vacuum diffusion pumps. G. O. Pritchard and G. H. Miller... 525
Salting-out chromatography: a review. William Rieman III..... 338
Saponification equivalents of phenolic esters, Determination of. S. W. Tobey, et al..... 611
"Sceptical Chymist," 1661. Roy G. Neville..... 106
Science Fair-International and the ACS Chemistry Awards, The National. Robert M. Brooker and Richard T. Rapala..... 209
Science fairs
Editorially speaking.....159, 277
Sciences and the humanities, The problem of the. Harold G. Cassidy..... 441
Semiconductors, Recent developments in. W. Crawford Dunlap..... 238
Separatory funnels, Substitute for. Marcel Jean..... 515
Shape of the 2*p* and related orbitals, The. Textbook errors, 28. Irwin Cohen... 20
Shield for small-scale apparatus, A convenient safety. Stanley H. Langer and Peter Pantages..... 634
Silver plating from an iodide complex. Tested demonstration..... A419
Simple demonstration of some precipitation and solubility effects. E. Matijevic, J. P. Kratohvil, and M. Kerker..... 397
Simple filtration technique for gravimetric determinations. Shirley W. Gaddis. 5
Simple method for cleaning buret tips, A. William Michels..... 548
Simple method for finding slopes. James P. Hoare..... 570
Simple test for nitric acid fumes. Alfred Chan, Jr..... 412
Simple test for the detection of phosphorus in organic compounds. Roger Ketcham and Anne de G. Low-Beer..... 414
Simplified moving-boundary transference cell. Stephen W. Tobey..... 516
Slopes, A simple method for finding. James P. Hoare..... 570
Solid sample identification analysis in high school chemistry. William Vasilakes..... 146
Solid state, Chemistry of the. Symposium. 224
Solid state, Electrochemistry of the. Allen B. Scott..... 317
Solid state inorganic materials, Recent advances in. Otto L. Forchheimer and Terry F. Newkirk..... 183
Solids and the gaseous corrosion of metals, Diffusion in. Walter J. Moore..... 232
Solubilities, molar, calculating from equilibrium constants. James N. Butler.. 460
Solubility effects, A simple demonstration of some precipitation and. E. Matijevic, et al..... 397
Solubility, Radioisotope demonstration of common ion effect on. E. Roig, et al. 350
Solubility, The effect of temperature on. Tested demonstration..... A99
Solutions, binary, alloys, gas adsorption, and ferromagnetism, Applications to. Elements of order-disorder theory. J. M. Honig..... 538
Solutions, Float method for the analysis of. James O. Osburn and Il C. Choi..... 578
Solutions, metal-in-amine, The nature of. E. Charles Evers..... 590
Solutions, volumetric, Apparatus for oxygen-sensitive. E. L. Martin and K. E. Bentley..... 264
Solvation and association from colligative properties by graphical means. George B. Smith, C. A. Hollingsworth, and Darl H. McDaniel..... 489
Sources, Convenient radioactivity. D. J. Carswell..... 352
Specific qualitative reagent for copper. T. R. Williams and Frederick Burton..... 413
Spectrometers, Infrared. Chemical instrumentation.....A5, A71
Spectroscopy, Inorganic infrared. John R. Ferraro..... 201
Spectroscopy, Nuclear magnetic resonance. John D. Roberts..... 581
Spring, Walther (1848–1911). (P) Jean Timmermans (Ralph E. Oesper, *Translator*)..... 422
Static thermodynamic properties, Total and. Edward A. Fletcher..... 257
Stereoisomerism of carbon compounds. William K. Noyce..... 23
Stirrers, A teflon bearing for. M. L. Kaplan, et al..... 577
Stoll, Arthur (P)..... 13
Strained ring double bonds, Frequency shifts in. Vibrating molecular models. Norman B. Colthup..... 394
Structural chemistry
Bonding..... 382
Dipole moments (TE)..... 522
Elementary course..... 530
Field theory..... 164
NMR spectroscopy..... 581
Rubber Stuart models..... 295
Stereoisomerism..... 23
Structure and spectra of nickel(II) and copper(II) complexes, The. W. Manch and W. Conard Fernelius... 192
Stuart-type molecular models, Inexpensive. William G. Hoover and Duward Shriver..... 295
Student experiment with filter paper electrophoresis. James E. Garvin..... 36
Sublimation. Tested demonstration..... A23
Substitute for separatory funnels. Marcel Jean..... 515
Substitution reactions of metal ions, Crystal field theory and. Ralph G. Pearson..... 164
Summer chemistry program for teenagers. Joseph K. Obold and Thomas S. Briggs..... 47
Summer institute, A pioneer. The G.E. Science Fellowship Program at Union College. E. K. Bacon..... 39
Summer institutes: Review and future plans. William E. Morrell..... 448
Summer research participation program for high school students, A. Reed F. Riley and C. G. Overberger..... 424
Surface area of activated charcoal by Langmuir adsorption isotherm. Boleslaw Ludwik Dunicz..... 357
Surface chemistry
Monomolecular films demonstration..... A751
Surface tension. Tested demonstration.... A99
Swarts, Frederic (1866–1940). (P) Jean Timmermans (Ralph E. Oesper, *Translator*)..... 422
Swarts, Theodor (P)..... 120
Symmetrical organic molecules, Dipole moments of. Textbook errors, 34. Wilbur N. Moulton..... 522
Symposia
Chemistry of the solid state.....224, 312
Cosmochemistry..... 52
Recent advances in inorganic chemistry.. 160
Synthesis of diamond, The. H. Tracy Hall..... 484
Synthesis of dimethylmaleate. Tested demonstration..... A179
Systematic way to avoid Le Chatelier's principle in chemical reactions. Lewis Katz..... 375
Systematization of inorganic cyclic compounds. I. Haiduc..... 134

Taste tells! Floyd L. James..... 291
Teacher training
G. E. institute at Union College..... 39
NSF institutes.....448, 451
Teaching machines
Programs for chemistry..... 463
Teaching techniques
Analog computer..... 492
Biochemistry..... 363
Colored slides..... 473
Developing concepts..... 433
Examinations..... 478
General chemistry content..... 255
Geometric approach ..123, (L)329, (E)329, 509
Modern analysis..... 455
No Le Chatelier's principle..... 375
Programmed instruction..... 463
Redox survey.....326, (L)637
Using filmed demonstrations..... 95
Technical writing
Jargon..... 415
Teenagers, A summer chemistry program for. Joseph K. Obold and Thomas S. Briggs..... 47
Teflon bearing for stirrers. M. L. Kaplan, R. E. Crocker, and Herbert Sargent 577
Temperature and power measurements in precision solution calorimetry. William F. O'Hara, Ching-Hsien Wu, and Loren G. Hepler..... 512

Terminology
Molar 549, 553, 555
Ternary diagrams. Textbook errors, 35. KAROL J. MYSELS 627
Tested demonstrations. FREDERIC B. DUTTON, *Editor.*
Cis-trans isomerization A179
Combining volumes: Hydrogen chloride and ammonia A813
Condensation of noxious gases A357
Dalton's law of partial pressures A545
A demonstration answer to a stereochemical puzzler A289
Demonstration notes: Burning phosphorus in air, catalytic oxidation of ammonia, color change with temperature, sublimation A23
Demonstration notes: Common ion effect; chlorine water substitute; rapid filtering without using a filter pump; writing on beakers, flasks, etc.; boiling liquid freezes water A813
Displacement reactions—spongy tin A637
The effect of temperature on solubility A99
Electrode potentials with a vacuum tube voltmeter A637
Fire under water A751
Graham's law of diffusion using overhead projector A23
Introduction to Le Chatelier's principle A491
Photochemical catalysis of an aliphatic substitution reaction A289
Preparation of metallic lithium A419
Qualitative demonstration of Raoult's law A545
Reaction to introduce Lewis acid-base chemistry, A A491
Silver plating from an iodide complex A419
Surface tension A99
Synthesis of dimethylmaleate A179
Transition temperature in iron A357
"Visible" monomolecular films: Estimation of molecular size and Avogadro's number A751
Textbook errors. Karol J. Mysels, *Editor.*
28. The shape of the 2*p* and related orbitals. IRWIN COHEN 20
29. The existence of endothermic adsorption. JOHN M. THOMAS 138
30. The ionization constant of water. THEDFORD P. DIRKSE 260
31. The pyrolytic decomposition of carboxylate salts to ketones. HARRY P. SCHULTE AND JACKSON P. SICHELS 300
32. The equilibria of complex formation. JAMES E. BANKS 391
33. The paddle-wheel Crookes tube. J. ARTHUR CAMPBELL 480
34. Dipole moments of symmetrical organic molecules. WILBUR N. MOULTON 522
35. Miscellanea No. 4: Chromyl chloride test for chlorides; "monovalent oxygen" compounds; ternary diagrams. K. J. MYSELS 627
Texts, general chemistry, Coordination chemistry in. ROY W. CLARK AND JOEL SELBIN 466
Theories on the origin of meteorites. EDWARD ANDERS AND GORDON G. GOLES 58
There are electrons in atoms! ANATOL SEMENTSOV 22
Thermoanalytical kinetic experiment: The catalyzed reaction of phenyl isocyanate with butanol. L. B. WEISFELD 88
Thermobalance, A controlled atmosphere. WESLEY W. WENDLANDT 566
Thermobalance for student experiments. RUDOLPH S. BOTTEI AND PAUL L. GERACE 568
Thermodynamic properties, Total and static. EDWARD A. FLETCHER 257
Thermodynamics
Bond energies 292
Diamond synthesis 484
Entropy 334
Gas dynamics 257
Gibbs-Duhem equation 509
Imperfect gases 140
Le Chatelier's principle 375
Order-disorder theory 538
Solid state 317
Three-dimensional representation of electrodeposition. W. H. REINMUTH 149
Tin(II), Benzenoid nitro-compounds as organic reagents for. ROBERT L. BURNS AND FRED M. GRUEN 410
Tin, spongy—Displacement reactions. Tested demonstration A637
Titration, A conductometric. Analysis of aspirin. JAMES S. PROCTOR AND JOHN E. ROBERTS 471
Titration controller for demonstrating the behavior of acid-base indicators. JOHN T. STOCK 633
Titrations with externally generated reagents, An inexpensive coulometer for. WILLIAM F. HEAD, JR. AND MAX M. MARSH 361
Total and static thermodynamic properties: Old gas dynamic properties which may be new to the chemist. EDWARD A. FLETCHER 257
Toxic reagents, Device for charging microtubes with. JOHN T. STOCK ET AL. 524
Training, graduate, of eminent chemists, Institutional influences in the. B. R. SIEBRING 630
Transference cell, Simplified moving-boundary. STEPHEN W. TOBEY 516
Transformer, A motor-driven variable. MEYER M. MARKOWITZ, ET AL. 96
Transition temperature in iron. Tested demonstration A357
Transulating and re-jargonising the little-languages of science. JACK DE MENT 415
Treasurer's report, Division of Chemical Education, ACS. WILLIAM G. KESSEL 635
Turbidimetric determination of lead, The. GERALD PERKINS, JR., J. W. WIMBERLEY, J. F. LAMB, AND L. E. MAURER 358
Ultra low conductivity water. National Bureau of Standards Summary Technical Report 421
Undergraduate training of chemistry majors: A report of the Bucknell Conference. BENNETT R. WILLEFORD, JR., AND LEALLYN B. CLAPP 251
Union College, The G.E. Science Fellowship Program at. A pioneer summer institute. E. K. BACON 39
Uses and abuses of bond energies. BRUCE E. KNOX AND HOWARD B. PALMER 292
Using filmed demonstrations in general chemistry. RUTH BOTDORF 95
Using the decay of Ba-137 to determine resolving time in G-M counting. ROBERT L. STEARNS AND JOSEPH F. MUCCI ..... 29, (L)329
Vacuum diffusion pumps, Safety device for. G. O. PRITCHARD AND G. H. MILLER 525
Vapor pressure apparatus, Isopiestic. G. W. DAILEY, ET AL. 28
Variable transformer, A motor-driven. MEYER M. MARKOWITZ, ET AL. 96
Vibrating molecular models: Frequency shifts in strained ring double bonds. NORMAN B. COLTHUP 394
Viscosities of butanol-heptane mixtures. K. KEITH INNES 564
"Visible" monomolecular films: Estimation of molecular size and Avogadro's number. Tested demonstration A751
Vitalism, vitality without. The Liebig-Pasteur controversy. GEORGE E. HEIN 614
Vitamin B_{12}, the deamidation of: Paper electrophoretic study of rates of consecutive reactions. GRANT H. BARLOW 37
Volumes, Combining: Hydrogen chloride and ammonia. Tested demonstration. A813
Walther Spring (1848–1911). (P) JEAN TIMMERMANS (RALPH E. OESPER, *Translator*) 422
Water by electrodialysis, Deionized. J. A. SCHUFLE 17
Water, The ionization constant of. Textbook errors, 30. THEDFORD P. DIRKSE 260
Water, Ultra low conductivity. National Bureau of Standards Summary Technical Report 421
Wieland, Heinrich (P) 15
Willstätter, Richard. (P) ROLF HUISGEN 10
Writing on beakers, flasks, etc. Tested demonstration A813

BOOKS REVIEWED

TITLES

About chemistry. MAGNUS PYKE 379
Abundance of chemical elements. V. V. CHERDYNTSEV 640
Advanced organic chemistry. G. W. WHELAND 221
Advances in fluorine chemistry. Vol. 1. M. STACEY, J. C. TATLOW, AND A. G. SHARPE, *Editors* A270
Advances in organic chemistry: Methods and results. Vols. 1 and 2. RALPH A. RAPHAEL, E. C. TAYLOR, AND HANS WYNBERG, *Editors* A438
Advances in protein chemistry. Vol. 14. C. B. ANFINSEN, JR., ET AL., *Editors* A54
AEDT y similares, Aplicaciones analíticas del. FRANCISCO BERMEJO MARTINEZ AND ANTONIO PRIETO BOUZA 274
Agricultural chemists, Official methods of analysis of. WILLIAM HORWITZ, *Editor.* 431
Alchemie zur Grossynthese, Von der. Grosse Chemiker. RUDOLF SACHTLEBEN AND ARMIN HERMANN 380
American Chemical Society specifications, 1960. Reagent chemicals. 1960 ACS Committee on Analytical Reagents A855
Amino acids, proteins, and cancer biochemistry. JOHN T. EDSALL, *Editor* A428
Ammonia, manufacture and uses. A. J. HARDING A196
Analysis, Chemical. Vol. 12: Systematic analysis of surface-active agents. MILTON J. ROSEN AND HENRY A. GOLDSMITH A52
Analysis, chemical, Physical methods in. Vol. 1. 2nd rev. ed. WALTER G. BERL, *Editor* 104
Analysis, inorganic, The use of organic reagents in. A. I. BUSEV AND N. G. POLIANSKII A922
Analysis of air pollutants, The chemical. MORRIS B. JACOBS A114

Analysis, Organic. Vol. 4. JOHN MITCHELL, JR., I. M. KOLTHOFF, E. S. PROSKAUER, AND A. WEISSBERGER, *Editors* A638
Analysis, Quantitative inorganic. R. BELCHER A44
Analytical chemical separations, Qualitative analysis and. PHILIP W. WEST AND MAURICE M. VICK A642
Analytical chemistry, Calculations of. LEICESTER F. HAMILTON AND STEPHEN G. SIMPSON 49
Analytical chemistry, Comprehensive. Vol. 1B, Classical analysis. CECIL L. WILSON AND DAVID W. WILSON A766
Analytical chemistry of the rare earths. R. C. VICKERY A768
Analytical chemistry, Treatise on. I. M. KOLTHOFF AND PHILIP J. ELVING. Part 1: Theory and Practice. Vol. 2..... A842
Part 2: Analytical chemistry of the elements. Vol. 1 ..... A844
Analytical methods, mechanisms and. Toxicology. C. P. STEWART AND A. STOLMAN, *Editors* A923
Anorganische Chemie. Band 1. ISTVAN NARAY-SZABO 380
Anorganischen chemie, Gmelins handbuch der. 8th ed. E. H. B. PIETSCH, *Editor*
System 15: Silicon, part C A432
System 33: Cadmium supplement A372
Anorganisches grundpraktikum. CARL MAHR A916
Aplicaciones analíticas del AEDT y similares. FRANCISCO B. MARTINEZ AND ANTONIO P. BOUZA 274
Atom, The book of the. LEONARD DE VRIES, *translated by* ERIC G. BREEZE 430
Atoms, molecules and chemical change. ERNEST GRUNWALD AND RUSSELL H. JOHNSEN 49
Atoms to stars, From. THEODORE A. ASHFORD 103

BIOCHEMISTRY, Comparative: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors*. Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
Biochemistry, Comparative, of photoreactive systems. MARY BELLE ALLEN, *Editor* 432
Biochemistry. Experimental. GERALD LITWACK 221
Biochemistry of plants and animals. M. FRANK MALLETTE, PAUL M. ALTHOUSE, AND CARL O. CLAGETT A282
Biochemistry, Radioactive isotopes in. ENGELBERT BRODA 536
Biology, Introduction to a submolecular. ALBERT SZENT-GYÖRGYI 276
Book of the atom, The. LEONARD DE VRIES, *translated by* ERIC G. BREEZE 430

CADMIUM supplement, System 33. Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. PIETSCH, *Editor* A372
Calculations of analytical chemistry. LEICESTER F. HAMILTON AND STEPHEN G. SIMPSON 49
Cancer biochemistry, Amino acids, proteins, and. JOHN T. EDSALL, *Editor* A428
Careers and opportunities in science. PHILIP POLLACK A636
Catalysis, heterogeneous, The mechanism of. J. H. DE BOER, *Editor* A494
Cavendish, Henry: His life and scientific work. A. J. BERRY A40
Ceramics, Introduction to. W. D. KINGERY A548
Chemical analysis. Vol. 12: Systematic analysis of surface-active agents. MILTON J. ROSEN AND HENRY A. GOLDSMITH A52
Chemical analysis of air pollutants, The. MORRIS B. JACOBS A114
Chemical change, Atoms, molecules, and. ERNEST GRUNWALD AND RUSSELL H. JOHNSEN 49
Chemical engineering practice. Vol. 10: Ancillary services. HERBERT W. CREMER AND SIDNEY B. WATKINS, *Editors*. 585
Chemical periodicity. R. T. SANDERSON .. 274
Chemical properties and identification of ions: An introduction to semi-micro qualitative analysis. OMER K. WHIPPLE A920
Chemical senses in health and disease, The. H. KALMUS AND S. J. HUBBARD A112
Chemical technology, Encyclopedia of (Edited by RAYMOND E. KIRK AND DONALD F. OTHMER). Second supplement volume. ANTHONY STANDEN, *Editor* A370
Chemistry. MICHELL J. SIENKO AND ROBERT A. PLANE 585
Chemistry for engineers. EDWARD CARTMELL 222
Chemistry of carbon compounds. Vol. 4. Part C: Heterocyclic compounds. E. H. RODD, *Editor* 588
Chemistry of lipids in health and disease. The. H. K. KING A284
Chemistry of nucleic acids, The. D. O. JORDAN 586
Chemistry of yttrium and scandium, The. R. C. VICKERY A268
Chimie minérale, Nouveau traité de. PAUL PASCAL, *Editor*. Vol. 7 ..... A116
Vol. 15 331
Clinical chemistry, Standard methods of. Vol. 3. DAVID SELIGSON, *Editor* 639
Cobalt: Its chemistry, metallurgy and uses. ROLAND S. YOUNG, *Editor* A498
College chemistry: A systematic approach. HARRY H. SISLER, CALVIN A. VANDERWERF, AND ARTHUR W. DAVIDSON ... A554
Colloid chemistry, Introduction to. KAROL J. MYSELS 50
Comparative biochemistry: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors*. Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
Comparative biochemistry of photoreactive systems. MARY BELLE ALLEN, *Editor* 432
Complex compounds, Instability constants of. K. B. YATSIMIRSKII AND V. P. VASIL'EV 157
Comprehensive analytical chemistry. Vol. 1B, Classical analysis. CECIL L. WILSON AND DAVID W. WILSON A766
Conservation, Teaching science through. MARTHA E. MUNZER AND PAUL F. BRANDWEIN 275
Crystal-structure analysis. M. J. BUERGER A364
Crystal structures. Supplement 5. W. G. WYCKOFF A770
Crystals and the polarizing microscope. N. H. HARTSHORNE AND A. STUART ... A121

DETONATION, Theory of. IA. B. ZELDOVICH AND A. S. KOMPANEETS 330
Dispensatory of the United States of America, The. New drug developments volume. ARTHUR OSOL, *Editor*. A926
Dispersion, Optical rotatory: Applications to organic chemistry. CARL DJERASSI. A48
Distillation, Molecular. G. BURROWS 536

EDUCATION, Policies, for science. FREDERICK L. FITZPATRICK, *Editor* 331
Einstein: A pictorial biography, WILLIAM CAHN 275
Electrolytic manufacture of chemicals from salt. D. W. F. HARDIE A196
Electronics for spectroscopists. C. G. CANNON, *Editor* A918
Electrophoresis in physiology. LENA A. LEWIS AND M. D. POULIK A52
Electrophoresis, The principles of. RENE AUDUBERT AND SERGE DE MENDE ... A52
Elements, Abundance of chemical. V V. CHERDYNTSEV 640
Elements, Analytical chemistry of the. Vol. 1 of Part 2 of Treatise on analytical chemistry. I. M. KOLTHOFF AND PHILIP J. ELVING A844
Encyclopedia of chemical technology (*Edited by* R. E. KIRK AND D. F. OTHMER). Second supplement vol. ANTHONY STANDEN, *Editor* A370
Engineering thermodynamics: An introductory textbook. JAMES B. JONES AND GEORGE A. HAWKINS A50
Engineers, Chemistry for. EDWARD CARTMELL 222
Experimental biochemistry. GERALD LITWACK 221
Experiments, chemistry, The golden book of. ROBERT BRENT A852
Experiments for instrumental methods. CHARLES N. REILLEY AND DONALD T. SAWYER 481
Experiments, Radioisotope, for the chemistry curriculum. NORMAN RADIN, *Editor* 379
FATTY acids: Their chemistry, properties, production, and uses. Part 1. KLARE S. MARKLEY, *Editor* A852
Fertility, The soil and its. H. TEUSCHER, R. ADLER, AND JEROME P. SEATON A286
Fluorine chemistry, Advances in. Vol. 1. M. STACEY, J. C. TATLOW, AND A. G. SHARPE, *Editors* A270
Food chemistry. LILLIAN HOAGLAND MEYER A202
Food processing, Nutritional evaluation of. ROBERT S. HARRIS AND HARRY VON LOESECKE, *Editors* A368
Free energy and biological function. Vol. 2 of Comparative biochemistry: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors* 158
From atoms to stars. THEODORE A. ASHFORD 103
Fuel cells. G. J. YOUNG, *Editor* 157

GENERAL chemistry. GARTH L. LEE AND HARRIS O. VAN ORDEN A110
General organic chemistry. WILLIAM C. STICKLER 534
Genetics, An outline of chemical. BERNARD S. STRAUSS A196
Geochemistry, Methods in. A. A. SMALES AND L. R. WAGNER, *Editors* A270
Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. PIETSCH, *Editor*.
System 15: Silicon, part C A432
System 33: Cadmium supplement A372
Golden book of chemistry experiments, The. ROBERT BRENT A852
Greek fire and gunpowder, A history of. J. R. PARTINGTON 430

HALOGENVERBINDUNGEN. Vol. 5, Part 4 of Methoden der organischen chemie. EUGENE MÜLLER, *Editor* A194
The Harvey Lectures. Series 54 222
Heat and thermodynamics. J. K. ROBERTS. 158
Henry Cavendish: His life and scientific work. A. J. BERRY A40
Heterocyclic compounds, An introduction to the chemistry of. R. M. ACHESON ... A430
Heterocyclic compounds, Part C of Vol. 4 of Chemistry of carbon compounds. E. H. RODD, *Editor* 588
History of chemistry, A short. J. R. PARTINGTON A374
History of Greek fire and gunpowder, A. J. R. PARTINGTON 430
History of metallography, A. CYRIL STANLEY SMITH 482

INORGANIC analysis, Quantitative. R. BELCHER A44

Inorganic chemistry: A guide to advanced study. R. B. Heslop and P. L. Robinson ... 331
Inorganic chemistry, Modern aspects of. H. J. Emeléus and J. S. Anderson ... 104
Inorganic chemistry, Progress in. Vol. 2. F. Albert Cotton, *Editor* ... A924
Inorganic chemistry, Synthetic. William L. Jolly ... 157
Inorganic chemistry, Textbook of. S. Young Tyree, Jr. and Kerro Knox ... 481
Inorganic syntheses. Vol. 6. Eugene G. Rochow, *Editor* ... A552
Insecticides, Modern, and world food production. F. A. Gunther and L. R. Jeppson ... A634
Instability constants of complex compounds. K. B. Yatsimirskii and V. P. Vasil'ev ... 157
Instrumental methods, Experiments for. Charles N. Reilley and Donald T. Sawyer ... 481
Introduction a l'etude des parfums. T. Bassiri ... A550
Introduction to a submolecular biology. Albert Szent-Györgyi ... 276
Introduction to ceramics. W. D. Kingery. A548
Introduction to colloid chemistry. Karol J. Mysels ... 50
Introduction to rubber technology. Maurice Morton, *Editor* ... 104
Introduction to solids. Leonid V. Azaroff. 586
Introduction to the chemistry of heterocyclic compounds. R. M. Acheson .. A430
Isotope effects on reaction rates. Lars Melander ... 330
Isotopes, Radioactive, in biochemistry. Engelbert Broda ... 536

Kinetics and mechanism. A. A. Frost and R. G. Pearson ... 535

Laboratory management and techniques. J. A. Edwards ... 587
Laboratory technique in organic chemistry. Kenneth B. Wiberg ... A192
Life, Viruses and the nature of. Wendell M. Stanley and Evans G. Valens ... 535
Lipids in health and disease, The chemistry of. H. K. King ... A284
Low-temperature techniques. F. Din and A. H. Cockett ... 275

Mechanism, Kinetics and. A. A. Frost and R. G. Pearson ... 535
Mechanism of heterogeneous catalysis, The. J. H. de Boer, *Editor* ... A494
Medicinal chemistry. Alfred Burger, *Editor* ... 276
Mental drugs: Chemistry's challenge to psychotherapy. O. A. Battista ... A550
Metallography, A history of. Cyril Stanley Smith ... 482
Metals and semiconductors, The surface chemistry of. Harry C. Gatos, *Editor* ... A548
Methoden der organischen chemie. Vol. 5, Part 4. Halogenverbindungen. Eugene Müller, *Editor* ... A194
Methods in geochemistry. A. A. Smales and L. R. Wagner, *Editors* ... A270
Microanalysis by the ring oven technique. Herbert Weisz ... 586
Microchemistry, Proceedings of the international symposium in ... A118
Micro-organisms, Polysaccharides of. M. Stacey and S. A. Barker ... 332
Modern aspects of inorganic chemistry. H. J. Emeléus and J. S. Anderson ... 104
Modern chemical processes. Vol. 6. Editors of *Industrial and Engineering Chemistry* ... A850
Modern insecticides and world food production. F. A. Gunther and L. R. Jeppson ... A634
Molecular distillation. G. Burrows ... 536
Molecular structure: The physical approach. J. C. D. Brand and J. C. Speakman ... 50
Molecules, and chemical change, Atoms. Ernest Grunwald and Russell H. Johnsen ... 49
Monographs for Teachers. Royal Institute of Chemistry, London, England ... 537

Name index of organic reactions. J. E. Gowan and T. S. Wheeler ... 588
Nouveau traitè de chimie minèrale. Paul Pascal, *Editor*. Vol. 7 ... A116
Vol. 15 ... 331
Nucleic acid outlines. Vol. 1. Van R. Potter ... A432
Nucleic acids, The. Vol. 3. Erwin Chargaff and J. N. Davidson, *Editors* ... A846
Nucleic acids, The chemistry of. D. O. Jordan ... 586
Nutritional evaluation of food processing. Robert S. Harris and Harry von Loesecke, *Editors* ... A368

Official methods of analysis of the Association of Official Agricultural Chemists. William Horwitz, *Editor* ... 431
Optical rotatory dispersion: Applications to organic chemistry. Carl Djerassi .. A48
Order, The search for. Cecil J. Schneer .. A108
Organic analysis. Vol. 4. John Mitchell, Jr., I. M. Kolthoff, E. S. Proskauer, and A. Weissberger, *Editors* ... A638
Organic analysis, Spot tests in. Fritz Feigl (*translated by* Ralph E. Oesper). A641
Organic chemistry. Allan R. Day and Madeleine M. Joullié ... 156
Organic chemistry. Keith M. Seymour .. 482
Organic chemistry, Advanced. G. W. Wheland ... 221
Organic chemistry, Advances in: Methods and results. Vols. 1 and 2. Ralph A. Raphael, E. C. Taylor, and Hans Wynberg, *Editors* ... A438
Organic chemistry, General. William C. Stickler ... 534
Organic chemistry, Laboratory technique in. Kenneth B. Wiberg ... A192
Organic chemistry, Physical methods of. Arnold Weissberger, *Editor*. Part 2 222
Parts 3 and 4 ... 432
Organic chemistry, Synthetic methods of. Vol. 14. W. Theilheimer ... 330
Organic chemistry, Tutorial questions in. P. A. Ongley ... A272
Organic compounds, Radiation chemistry of. A. J. Swallow ... 640
Organic reactions, Name index of. J. E. Gowan and T. S. Wheeler ... 588
Organic reactions. Vol. 11. Arthur C. Cope, *Editor-in-chief* ... 431
Organic reagents in inorganic analysis, The use of. A. I. Busev and N. G. Polianskii ... A923
Organic syntheses. Vol. 40. Melvin S. Newman, *Editor* ... A444
Organic systems, Oxidation-reduction potentials of. W. Mansfield Clark ... 158
Organischen chemie, Methoden der. Vol. 5, Part 4: Halogenverbindungen. Eugene Müller, *Editor* ... A194
Organometallic chemistry. H. Zeiss, *Editor* ... 587
Outline of chemical genetics. Bernard S. Strauss ... A196
Oxidation-reduction potentials of organic systems. W. Mansfield Clark ... 158

Parfums, Introduction a l'etude des. T. Bassiri ... A550
Periodicity, Chemical. R. T. Sanderson. 274
Pharmacy, Physical. Alfred N. Martin .. A121
Phosphorus esters, Toxic. Richard D. O'Brien ... 380
Photochemistry in the liquid and solid states. Lawrence J. Heidt, Robert S. Livingston, Eugene Rabinowitch, and Farrington Daniels, *Editors* ... A276
Photoreactive systems, Comparative biochemistry of. Mary Belle Allen, *Editor* ... 432
Physical approach, The: Molecular structure. J. C. D. Brand and J. C. Speakman ... 50
Physical chemistry. Farrington Daniels and Robert A. Alberty ... 639
Physical chemistry of surfaces. Arthur W. Adamson ... A366
Physical methods in chemical analysis. Vol. 1. 2nd rev. ed. Walter G. Berl, *Editor* ... 104
Physical methods of organic chemistry. Arnold Weissberger, *Editor*. Part 2. 222
Parts 3 and 4 ... 432
Physical pharmacy. Alfred N. Martin .. A121
Physical science. Donald S. Allen and Richard J. Ordway ... 156
Physico-chemical constants of binary systems in concentrated solutions. Vol. 4: Systems with inorganic plus organic or inorganic compounds (excepting metallic derivatives). Jean Timmermans. A644
Physiology, Electrophoresis in. Lena A. Lewis and M. D. Poulik ... A52
Plastic, Source book of the new. Vol. 2. Herbert R. Simonds ... A855
Polarising microscope, Crystals and the. N. H. Hartshorne and A. Stuart ... A121
Policies for science education. Frederick L. Fitzpatrick, *Editor* ... 331
Pollutants, The chemical analysis of air. Morris B. Jacobs ... A114
Polymer chemistry, Preparative methods of. Wayne Sorenson and Tod W. Campbell ... A840
Polymers, Properties and structure of. A. V. Tobolsky ... A922
Polysaccharides of micro-organisms. M. Stacey and S. A. Barker ... 332
Preparative methods of polymer chemistry. Wayne Sorenson and Tod W. Campbell ... A840
Principles of chemical thermodynamics. Charles E. Reid ... 50
Principles of electrophoresis, The. Rene Audubert and Serge de Mende ... A52
Proceedings of the International Symposium in Microchemistry ... A118
Process of education, The. Jerome S. Bruner ... A276
Processes, Modern chemical. Vol. 6. Editors of *Industrial and Engineering Chemistry* ... A850
Programmed learning, Teaching machines and. A. A. Lumsdaine and Robert Glaser, *Editors* ... 275
Progress in inorganic chemistry. Vol. 2. F. Albert Cotton, *Editor* ... A924
Properties and structure of polymers. A. V. Tobolsky ... A922
Protein chemistry, Advances. Vol. 14. C. B. Anfinsen, Jr., et al., *Editors* ... A54
Proteins, and cancer biochemistry, Amino acids. John T. Edsall, *Editor* ... A428
Psychotherapy, chemistry's challenge to. Mental drugs. O. A. Battista ... A550

Qualitative analysis, an introduction to semi-micro: Chemical properties and identification of ions. Omer K. Whipple ... A920
Qualitative analysis and analytical chemical separations. Philip W. West and Maurice M. Vick ... A642
Quantitative inorganic analysis. R. Belcher ... A44
Quantum chemistry: Methods and applications. R. Daudel, R. Lefebvre, and C. Moser ... 103

Radiation chemistry of organic compounds. A. J. Swallow ... 640
Radioactive isotopes in biochemistry. Engelbert Broda ... 536

Radiochemistry of the rare earths, scandium, yttrium and actinium, The. P. C. Stevenson and W. E. Nervik..... A768
Radioisotope experiments for the chemistry curriculum. Norman Radin, *Editor*.. 379
Radioisotope techniques. Ralph T. Overman and Herbert M. Clark..... A436
Rare earths, Analytical chemistry of the. R. C. Vickery..... A768
Rare earths, scandium, yttrium, and actinium, The radiochemistry of the. P. C. Stevenson and W. E. Nervik.. A768
Rates, Isotope effects on reaction. Lars Melander..... 330
Reagent chemicals. ACS specifications, 1960. 1960 ACS Committee on Analytical Reagents..... A855
Restless atom, The. Alfred Romer. The Science study series. Vol. 12..... 588
Ring oven technique, Microanalysis by the. Herbert Weisz..... 586
Rubber technology, Introduction to. Maurice Morton, *Editor*..... 104
Russian, Scientific. James W. Perry..... 535
Russian for scientists. C. R. Buxton and H. Sheldon Jackson..... A500

Salt, Electrolytic manufacture of chemicals from. D. W. F. Hardie..... A196
Scandium, The chemistry of yttrium and. R. C. Vickery..... A268
Science education, Policies for. Frederick L. Fitzpatrick, *Editor*..... 331
Science study series. Vol. 12. The restless atom. Alfred Romer..... 588
Vol. 18. Water, the mirror of science. Kenneth S. Davis and John A. Day. 588
Scientific Russian. James W. Perry..... 535
Search for order, The. Cecil J. Schneer. A108
Selective toxicity. Adrien Albert..... 332
Semiconductors, The surface chemistry of metals and. Harry C. Gatos, *Editor*. A548
Semi-micro qualitative analysis, An introduction to: Chemical properties and identification of ions. Omer K. Whipple..... A920
Senses, The chemical, in health and disease. H. Kalmus and S. J. Hubbard..... A112
Separation processes in practice. Robert F. Chapman, *Editor*..... 482
Short history of chemistry. J. R. Partington..... A374
Silicon, Part C, System 15. Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. Pietsch, *Editor*..... A432
Soil and its fertility, The. H. Teuscher, R. Adler and Jerome P. Seaton.... A286
Solids, Introduction to. Leonid V. Azároff..... 586
Source book of the new plastic. Vol. 2. Herbert R. Simonds..... A855
Spectrochemical analysis. L. H. Ahrens and S. R. Taylor..... A644
Spot tests in organic analysis. Fritz Feigl (*translated by* Ralph E. Oesper)..... A641
Standard methods of clinical chemistry. Vol. 3. David Seligson, *Editor*..... 639
Statistical manual for chemists. Edward L. Bauer..... A46
Statistical theory and methodology in science and engineering. K. A. Brownlee..... A370
Story of chemistry, The. Georg Lockemann..... A116
Structure of science, A. Joseph H. Simons..... 276
Style guide for chemists. Louis F. Fieser and Mary Fieser..... A440
Substitution at elements other than carbon. C. K. Ingold..... A434
Surface-active agents, Systematic analysis of. Chemical analysis. Vol. 12. Milton J. Rosen and Henry A. Goldsmith..... A82
Surface chemistry of metals and semiconductors, The. Harry C. Gatos, *Editor*..... A548
Surfaces, Physical chemistry of. Arthur W. Adamson..... A366
Syntheses, Inorganic. Vol. 6. Eugene G. Rochow, *Editor*..... A552
Syntheses, Organic. Vol. 40. Melvin S. Newman, *Editor*..... A444
Synthetic inorganic chemistry. William L. Jolly..... 157
Synthetic methods of organic chemistry. Vol. 14. W. Theilheimer..... 330

Tables of thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam. Joseph Hilsenrath, et al..... 536
Teaching machines and programmed learning. A. A. Lumsdaine and Robert Glaser, *Editors*..... 275
Teaching science through conservation. Martha E. Munzer and Paul F. Brandwein..... 275
Ternary systems. G. Masing (B. A. Rogers, *Translator*)..... A848
Textbook of chemistry. Stella Goostray and J. Rae Schwenck..... A766
Textbook of inorganic chemistry. S. Young Tyree, Jr. and Kerro Knox. 481
Theorie fondamentale du system periodique des elements. Stanislas Dockx..... 222
Theory of detonation. Ia. B. Zeldovich and A. S. Kompaneets..... 330
Thermal engineering. Harry L. Solberg, Orville C. Cromer, and Albert R. Spalding..... A204
Thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam, Tables of. Joseph Hilsenrath, et al..... 536
Thermodynamics, Engineering: An introductory textbook. James B. Jones and George A. Hawkins..... A50
Thermodynamics, Heat and. J. K. Roberts..... 158
Thermodynamics, Principles of chemical. Charles E. Reid..... 50
Thermoelectricity. Paul H. Egli, *Editor*. 534
Toxic phosphorus esters. Richard D. O'Brien..... 380
Toxicity, Selective. Adrien Albert..... 332
Toxicology: Mechanisms and analytical methods. C. P. Stewart and A. Stolman, *Editors*..... A923
Transport phenomena. R. Byron Bird, Warren E. Stewart, and Edwin N. Lightfoot..... A640
Transport and thermodynamic properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam, Tables of. Joseph Hilsenrath, et al..... 536
Treatise on analytical chemistry. I. M. Kolthoff and Philip J. Elving. Part 1: Theory and practice. Vol. 2.. A842
Part 2: Analytical chemistry of the elements. Vol. 1..... A844
Tutorial questions in organic chemistry. P. A. Ongley..... A272

Ultrafiltration. L. Ambard and S. Trautmann..... 588
Use of organic reagents in inorganic analysis. A. I. Busev and N. G. Polianskii... A923

Valency, Wave mechanics and. J. W. Linnett..... 431
Viruses and the nature of life. Wendell M. Stanley and Evans G. Valens... 535
Von der Alchemie zur Grossynthese. Grosse Chemiker. Rudolf Sachtleben and Armin Hermann..... 380

Water, the mirror of science. Kenneth S. Davis and John Arthur Day. The Science study series. Vol. 18..... 588
Wave mechanics and valency. J. W. Linnett..... 431

X-ray absorption and emission in analytical chemistry. H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, and P. D. Zemany..... 430

Yttrium and scandium, The chemistry of. R. C. Vickery..... A268

AUTHORS

Acheson, R. M. An introduction to the chemistry of heterocyclic compounds.. A430
ACS, 1960 Committee on Analytical Reagents. Reagent chemicals. American Chemical Society specifications, 1960..... A855
Adamson, Arthur W. Physical chemistry of surfaces..... A366
Ahrens, L. H., and Taylor, S. R. Spectrochemical analysis..... A644
Albert, Adrien. Selective toxicity..... 332
Alberty, Robert A.—*See* Daniels, Farrington
Allen, Donald S., and Ordway, Richard J. Physical science..... 156
Allen, Mary Belle, *Editor*. Comparative biochemistry of photoreactive systems..... 432
Althouse, Paul M.—*See* Mallette, M. Frank
Ambard, L., and Trautmann, S. Ultrafiltration..... 588
Anfinsen, C. B., Jr., Anson, M. L., Bailey, Kenneth, and Edsall, John T., *Editors*. Advances in protein chemistry. Vol. 14..... A54
Anderson, J. S.—*See* Emeléus, H. J.
Anson, M. L.—*See* Anfinsen, C. B., Jr.
Ashford, Theodore A. From atoms to stars..... 103
Audubert, Rene and de Mende, Serge. The principles of electrophoresis..... A52
Azaroff, Leonid V. Introduction to solids..... 586

Bailey, Kenneth—*See* Anfinsen, C. B., Jr.
Barker, S. A.—*See* Stacey, M.
Bassiri, T. Introduction a l'etude des parfums..... A550
Battista, O. A. Mental drugs: Chemistry's challenge to psychotherapy.... A550
Bauer, Edward L. A statistical manual for chemists..... A46
Belcher, R. Quantitative inorganic analysis..... A44
Berl, Walter G., *Editor*. Physical methods in chemical analysis. Vol. 1. 2nd rev. ed..... 104
Berry, A. J. Henry Cavendish: His life and scientific work..... A40
Bird, R. Byron, Stewart, Warren E., and Lightfoot, Edwin N. Transport phenomena..... A640
Bouza, Antonio—*See* Martinez, Francisco
Brand, J. C. D. and Speakman, J. C. Molecular structure: The physical approach..... 50
Brandwein, Paul F.—*See* Munzer, Martha E.
Breese, Eric G., *Translator*. The book of the atom, by Leonard de Vries.
Brent, Robert. The golden book of chemistry experiments..... A852
Broda, Engelbert. Radioactive isotopes in biochemistry..... 536
Brownlee, K. A. Statistical theory and methodology in science and engineering..... A370
Bruner, Jerome S. The process of education..... A276
Buerger, M. J. Crystal-structure analysis..... A364

BURGER, ALFRED, *Editor.* Medicinal chemistry 276
BURROWS, G. Molecular distillation 536
BUSEV, A. I., AND POLIANSKII, N. G. The use of organic reagents in inorganic analysis A923
BUXTON, C. R., AND JACKSON, H. SHELDON. Russian for scientists A500

CAHN, WILLIAM. Einstein: A pictorial biography 275
CAMPBELL, TOD W.—*See* SORENSON, WAYNE
CANNON, C. G., *Editor.* Electronics for spectroscopists A918
CARTMELL, EDWARD. Chemistry for engineers 222
CHAPMAN, ROBERT F., *Editor.* Separation processes in practice 482
CHARGAFF, ERWIN, AND DAVIDSON, J. N., *Editors.* The nucleic acids.—Vol. 3 .. A846
CHERDYNTSEV, V. V. Abundance of chemical elements 640
CLAGETT, CARL O.—*See* MALLETTE, M. FRANK
CLARK, HERBERT M.—*See* OVERMAN, RALPH T.
CLARK, W. MANSFIELD. Oxidation-reduction potentials of organic systems. 158
COCKETT, A. H.—*See* DIN, F.
COPE, ARTHUR C., *Editor.* Organic reactions. Vol. 11 431
COTTON, F. ALBERT, *Editor.* Progress in inorganic chemistry. Vol. 2 ..... A924
CREMER, HERBERT W., AND WATKINS, SIDNEY B., *Editors.* Chemical engineering practice. Vol. 10: Ancillary services 585
CROMER, ORVILLE C.—*See* SOLBERG, HARRY L.

DANIELS, FARRINGTON, AND ALBERTY, ROBERT A. Physical chemistry 639
—*See* HEIDT, LAWRENCE J.
DAUDEL, R., LEFEBVRE, R. AND MOSER, C. Quantum chemistry: Methods and applications 103
DAVIDSON, ARTHUR W.—*See* SISLER, HARRY H.
DAVIDSON, J. N.—*See* CHARGAFF, ERWIN
DAVIS, KENNETH S., AND DAY, JOHN ARTHUR. Water, the mirror of science. Science study series. Vol. 18 588
DAY, ALLAN R. AND JOULLIÉ, MADELEINE M. Organic chemistry 156
DAY, JOHN ARTHUR—*See* DAVIS, KENNETH S.
DE BOER, J. H., *Editor.* The mechanism of heterogeneous catalysis A494
DE MENDE, SERGE—*See* AUDUBERT, RENE
DE VRIES, LEONARD. The book of the atom. (*Translated by* ERIC G. BREEZE) 430
DIN, F. AND COCKETT, A. H. Low-temperature techniques 275
DJERASSI, CARL. Optical rotatory dispersion: Applications to organic chemistry A48
DOCKX, STANISLAS. Theorie fondamentale du system periodique des elements 222

EDITORS of *Industrial and Engineering Chemistry.* Modern chemical processes. Vol. 6 ..... A850
EDSALL, JOHN T., *Editor.* Amino acids, proteins, and cancer biochemistry A428
—*See* ANFINSEN, C. B., JR
EDWARDS, J. A. Laboratory management and techniques 557
EGLI, PAUL H., *Editor.* Thermoelectricity. 534
ELVING, PHILIP J.—*See* KOLTHOFF, I. M.
EMELÉUS, H. J., AND ANDERSON, J. S. Modern aspects of inorganic chemistry. 104

FEIGL, FRITZ (*translated by* OESPER, RALPH E.). Spot tests in organic analysis ... A841

FIESER, LOUIS F., AND FIESER, MARY. Style guide for chemists A440
FIESER, MARY—*See* FIESER, LOUIS F.
FITZPATRICK, FREDERICK L., *Editor.* Policies for science education 331
FLORKIN, MARCEL, AND MASON, HOWARD S., *Editors.* Comparative biochemistry: A comprehensive treatise.
Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
FROST, A. A., AND PEARSON, R. G. Kinetics and mechanism 535

GATOS, HARRY C., *Editor.* The surface chemistry of metals and semiconductors A548
GLASER, ROBERT—*See* LUMSDAINE, A. A.
GOLDSMITH, HENRY A.—*See* ROSEN, MILTON J.
GOOSTRAY, STELLA, AND SCHWENCK, J. RAE. A textbook of chemistry A766
GOWAN, J. E., AND WHEELER, T. S. Name index of organic reactions 588
GRUNWALD, ERNEST, AND JOHNSEN, RUSSELL H. Atoms, molecules and chemical change 49
GUNTHER, F. A., AND JEPPSON, L. R. Modern insecticides and world food production A634

HAMILTON, LEICESTER F., AND SIMPSON, STEPHEN G. Calculations of analytical chemistry 49
HARDIE, D. W. F. Electrolytic manufacture of chemicals from salt A196
HARDING, A. J. Ammonia, manufacture and uses A196
HARRIS, ROBERT S., AND LOESECKE, HARRY VON, *Editors.* Nutritional evaluation of food processing A368
HARTSHORNE, N. H. AND STUART, A. Crystals and the polarising microscope A121
HAWKINS, GEORGE A.—*See* JONES, JAMES B.
HEIDT, LAWRENCE J., LIVINGSTON, ROBERT S., RABINOWITCH, EUGENE, AND DANIELS, FARRINGTON, *Editors.* Photochemistry in the liquid and solid states A276
HERMANN, ARMIN—*See* SACHTLEBEN, RUDOLF
HESLOP, R. B. AND ROBINSON, P. L. Inorganic chemistry: A guide to advanced study 331
HILSENRATH, JOSEPH, ET AL. Tables of thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam 536
HORWITZ, WILLIAM, *Editor.* Official methods of analysis of the Association of Official Agricultural Chemists 431
HUBBARD, S. J.—*See* KALMUS, H.

INGOLD, C. K. Substitution at elements other than carbon A434

JACKSON, H. SHELDON—*See* BUXTON, C. R.
JACOBS, MORRIS B. The chemical analysis of air pollutants A114
JEPPSON, L. R.—*See* GUNTHER, F. A.
JOHNSEN, RUSSELL H.—*See* GRUNWALD, ERNEST
JOLLY, WILLIAM L. Synthetic inorganic chemistry 157
JONES, JAMES B. AND HAWKINS, GEORGE A. Engineering thermodynamics: An introductory textbook A50
JORDAN, D. O. The chemistry of nucleic acids 586
JOULLIÉ, MADELEINE M.—*See* DAY, ALLAN R.

KALMUS, H. AND HUBBARD, S. J. The chemical senses in health and disease .. A113
KING, H. K. The chemistry of lipids in health and disease A284
KINGERY, W. D. Introduction to ceramics. A548
KIRK, RAYMOND E. AND OTHMER, DONALD F. Encyclopedia of chemical technology. Second supplement volume, Edited by STANDEN, ANTHONY A370
KNOX, KERRO—*See* TYREE, S. YOUNG, JR.
KOLTHOFF, I. M., AND ELVING, PHILIP J. Treatise on analytical chemistry. Part 1: Theory and practice. Vol. 2 .. A842
Part 2: Analytical chemistry of the elements. Vol. 1 ..... A844
—*See* MITCHELL, JOHN, JR.
KOMPANEETS, A. S.—*See* ZELDOVICH, IA. B.

LEE, GARTH L., AND VAN ORDEN, HARRIS O. General chemistry A110
LEFEBVRE, R.—*See* DAUDEL, R.
LEWIS, LENA A., AND POULIK, M. D. Electrophoresis in physiology A52
LIEBHAFSKY, H. A., PFEIFFER, H. G., WINSLOW, E. H., AND ZEMANY, P. D. X-ray absorption and emission in analytical chemistry 430
LIGHTFOOT, EDWIN N.—*See* BIRD, R. BYRON
LINNETT, J. W. Wave mechanics and valency 431
LITWACK, GERALD. Experimental biochemistry 221
LIVINGSTON, ROBERT S.—*See* HEIDT, LAWRENCE J.
LOCKEMANN, GEORG. The story of chemistry A116
LUMSDAINE, A. A. AND GLASER, ROBERT, *Editors.* Teaching machines and programmed learning 275

MAHR, CARL. Anorganisches grund praktikum A916
MALLETTE, M. FRANK, ALTHOUSE, PAUL M., AND CLAGETT, CARL O. Biochemistry of plants and animals A282
MARKLEY, KLARE S., *Editor.* Fatty acids: Their chemistry, properties, production, and uses. Part 1 ..... A852
MARTIN, ALFRED N. Physical pharmacy .. A121
MARTINES, FRANCISCO B. AND BOUSA, ANTONIO. Aplicaciones analíticas del AEDT y similares 274
MASING, G. (ROGERS, B. A., *Translator*). Ternary systems A848
MASON, HOWARD S.—*See* FLORKIN, MARCEL
MELANDER, LARS. Isotope effects on reaction rates 330
MEYER, LILLIAN HOAGLAND. Food chemistry A202
MITCHELL, JOHN JR., KOLTHOFF, I. M., PROSKAUER, E. S., AND WEISSBERGER, A., *Editors.* Organic analysis. Volume 4 ..... A638
MORTON, MAURICE, *Editor.* Introduction to rubber technology 104
MOSER, C.—*See* DAUDEL, R.
MÜLLER, EUGENE, *Editor.* Methoden der organischen chemie. Vol. 5, Part 4: Halogenverbindungen A194
MUNSER, MARTHA E. AND BRANDWEIN, PAUL F. Teaching science through conservation 275
MYSELS, KAROL J. Introduction to colloid chemistry 50

NARAY-SZABO, ISTVAN. Anorganische chemie. Vol. 1 380
NERVIK, W. E.—*See* STEVENSON, P. C.
NEWMAN, MELVIN S., *Editor.* Organic syntheses. Vol. 40 ..... A444

O'BRIEN, RICHARD D. Toxic phosphorus esters 380
ONGLEY, P. A. Tutorial questions in organic chemistry A272
ORDWAY, RICHARD J.—*See* ALLEN, DONALD S.
OPEL, ARTHUR, *Editor.* The dispensatory of the United States of America. New drug development volume A380

Nevin 144
Langmuir adsorption isotherm, Surface area of activated charcoal by. Boleslaw Ludwik Dunicz 357
Latin America, Chemical information centers of. J. Vikin 628
Le Chatelier's principle in chemical reactions, A systematic way to avoid. Lewis Katz 375
Le Chatelier's principle, Introduction to. Tested demonstration A491
Lead, The turbidimetric determination of. Gerald Perkins, Jr., et al. 358
Lecture demonstrations
Optical rotation 263
Precipitation and solubility 397
Precipitation from homogeneous solution. 16
Stable carbanions 307
Lewis acid-base chemistry. A reaction to introduce. Tested demonstration ... A491
Liebig birthplace memorial. Ralph E. Oesper 122
Liebig-Pasteur controversy, The: Vitality without vitalism. George E. Hein .. 614
Ligand field theory—See crystal field theory
Structure and spectra 192
Lithium, preparation of metallic. Tested demonstration A419
Little-languages of science, Translating and re-jargonizing the. Jack De Ment ... 415
Low cost direct current source for the laboratory. Robert Barnard and Ray Woodriff 521
Luminescence of inorganic crystals, The. Fred E. Williams 242

Machine-sorted punched card systems, Adapting to hand sorting and coding. Stanley Kirschner 526
Models, Vibrating molecular: Frequency shifts in strained ring double bonds. Norman B. Colthup 394
Mole
Editorially speaking 51
Collected comments 549
"Mole," A redefinition of. Shiu Lee 549
Mole and related quantities, The. E. A. Guggenheim ..... 86, (E) 131, (L) 554
Mole in quantitative chemistry, The. George Novello Copley 551
Molecular models featuring molecular orbitals. George C. Brumlik 502
Molecular models, Inexpensive Stuart-type. William G. Hoover and Duward Shriver 295
Molecular models, Vibrating: Frequency shifts in strained ring double bonds. Norman B. Colthup 394
Molecular oribitals, Molecular models featuring. George C. Brumlik 502
Molecular structure
Bonding 382
Molecular weights by cryoscopy. Robert Mikular and Olaf Runquist 537
Moles and equivalents: Quantities of matter. Irwin Cohen 555
Monomolecular films, "Visible": Estimation of molecular size and Avogadro's number. Tested demonstration A751
"Monovalent oxygen" compounds. Textbook errors, 35. Karol J. Mysels ... 627
Montana State Conference, A report of the: The college chemistry course for non-science majors. A. B. Garrett, et al. 253
Motor-driven variable transformer, A. Meyer M. Markowitz, Daniel A. Bortta, and George Capriola 96
Museums
Franklin Institute 597
organic chemistry. J. C. Martin 286
Nomenclature, organic, The general philosophy of. Charles D. Hurd 43
Nominations for office of chairman-elect, 1961–62. Division of Chemical Education, ACS 273
Nomographs for first and second order kinetics. S. G. Smith and I. D. R. Stevens 574
Non-cyanide detection of cadmium in the presence of copper. Ramesh Chandra ..... 409, (L) 638
Non-metallic compounds, Non-stoichiometry in. Leo Brewer 90
Non-science majors, The college chemistry course for. A. B. Garrett, W. B. Cook, and W. T. Lippincott 253
Non-stoichiometry in non-metallic compounds. Leo Brewer 90
Notes on qualitative analysis. A Chem Ed Collection 406
Nuclear chemistry
Nucleogenesis 67
Nuclear magnetic resonance spectroscopy. John D. Roberts 581
Nuclear radiation detectors. Chemical instrumentation. S. Z. Lewin A135
Nuclear radiation electronic gear. Chemical instrumentation. S. Z. Lewin A225, A305, A389, A457
Nucleosynthesis and extinct natural radioactivity, Chronology of. Truman P. Kohman 73

Optical rotation, A model for. Leon L. Jones and Henry Eyring 601
Optical rotation with an "Eskimo yo-yo," A demonstration of. S. J. Gill 263

Ratio errors in pseudo first order reactions. Fred Sicilio and M. D. Peterson ... 576
Reaction kinetics by differential thermal analysis. Wesley W. Wendlandt ... 571
Reaction to introduce Lewis acid-base chemistry. Tested demonstration A491
Reactive intermediates of organic chemistry. Ross Stewart 308
Recent advances in inorganic chemistry. Symposium. Introduction. R. W. Parry 160
Recent advances in solid state inorganic materials. Otto L. Forchheimer and Terry F. Newkirk 183
Recent developments in metal carbonyl synthesis. Harold E. Podall 187
Recent developments in N—F chemistry. Charles B. Colburn 180
Recent developments in semiconductors. W. Crawford Dunlap 238
Recent improvements in our knowledge of cosmic abundances. William D. Ehmann 53
Redefinition of "Mole," A. Shiu Lee 549
Redetermination of the half-life of carbon-14. National Bureau of Standards Office of Technical Information 82
Redox revisited. Karl L. Lockwood ..... 326, (L) 637
Reducing agents, New selective. Herbert C. Brown 173
Reducing certain metallic isotopic oxides, A rapid efficient method for. Philip S. Baker 117
Re-jargonizing the little-languages of science, Translating and. Jack De Ment 415
Research ideas for young chemists—*See Chemical projects*
Research participation program for high school students, A summer. Reed F. Riley and C. G. Overberger 424
Semiconductors, Recent developments in. W. Crawford Dunlap 238
Separatory funnels, Substitute for. Marcel Jean 515
Shape of the 2*p* and related orbitals, The. Textbook errors, 28. Irwin Cohen ... 20
Shield for small-scale apparatus, A convenient safety. Stanley H. Langer and Peter Rantages 634
Silver plating from an iodide complex. Tested demonstration A419
Simple demonstration of some precipitation and solubility effects. E. Matijevic, J. P. Kratohvil, and M. Kerker 397
Simple filtration technique for gravimetric determinations. Shirley W. Gaddis. 5
Simple method for cleaning buret tips, A. William Michels 548
Simple method for finding slopes. James P. Hoare 570
Simple test for nitric acid fumes. Alfred Chan, Jr. 412
Simple test for the detection of phosphorus in organic compounds. Roger Ketcham and Anne de G. Low-Beer 414
Simplified moving-boundary transference cell. Stephen W. Tobey 516
Slopes, A simple method for finding. James P. Hoare 570
Solid sample identification analysis in high school chemistry. William Vasilakes 146
Solid state, Chemistry of the. Symposium. 224
Solid state, Electrochemistry of the. Allen B. Scott 317
Solid state inorganic materials, Recent advances in. Otto L. Forchheimer and Terry F. Newkirk 183
Solids and the gaseous corrosion of metals, Diffusion in. Walter J. Moore 232
Solubilities, molar, calculating from equi-
Structural chemistry
Bonding 382
Dipole moments (TE) 522
Elementary course 530
Field theory 164
NMR spectroscopy 581
Rubber Stuart models 295
Stereoisomerism 23
Structure and spectra of nickel(II) and copper(II) complexes, The. W. Manch and W. Conard Fernelius ... 192
Stuart-type molecular models, Inexpensive. William G. Hoover and Duward Shriver 295
Student experiment with filter paper electrophoresis. James E. Garvin 36
Sublimation. Tested demonstration A23
Substitute for separatory funnels. Marcel Jean 515
Substitution reactions of metal ions, Crystal field theory and. Ralph G. Pearson 164
Summer chemistry program for teenagers Joseph K. Obold and Thomas S. Briggs 47
Summer institute, A pioneer. The G.E. Science Fellowship Program at Union College. E. K. Bacon 39
Summer institutes: Review and future plans. William E. Morrell 448
Summer research participation program for high school students, A. Reed F. Riley and C. G. Overberger 424
Surface area of activated charcoal by Langmuir adsorption isotherm. Boleslaw Ludwik Dunicz 357
Surface chemistry
Monomolecular films demonstration A751
Surface tension. Tested demonstration A99
Swarts, Frederic (1866–1940). (P) Jean Timmermans (Ralph E. Oesper, *Translator*) 422

compounds as. Robert L. Burns and Fred M. Gruen ... 410
Origin of meteorites, Theories on the. Edward Anders and Gordon G. Goles ... 58
Overhead projector, Graham's law of diffusion using. Tested demonstration ... A23
Oxidation-reduction mechanisms. Frederick R. Duke ... 161
Oxygen-sensitive volumetric solutions, Apparatus for. E. L. Martin and K. E. Bentley ... 264

Paddle-wheel Crookes tube, The. Textbook errors, 33. J. Arthur Campbell 480
Paper chromatographic scheme for the identification of metallic ions, A. A. S. Ritchie ... 400
Paper chromatography, Demonstrating enzymatic hydrolysis of glucosides by. S. F. Darling ... 508
Paper chromatography, Ion exchange. Alan T. Thomas and J. P. Phillips ... 406
Paper electrophoretic study of rates of consecutive reactions: The deamidation of vitamin B12. Grant H. Barlow ... 37
Paper-made crystal models. Yasuyuki Komuro and Kôzô Sone ... 580
Paramagnetic, Electron, resonance of free radicals. Juan A. McMillan ... 438
Partial pressures, Dalton's law of. Tested demonstration ... A545
Pasteur-Liebig controversy: Vitality without vitalism. George E. Hein ... 614
Patterson, Robert Maskell: First to teach atomic theory in America? (P) Wyndham D. Miles ... 561
Metal oxide theory ... 9[illegible]
Molecular vibrations ... 394
NMR spectroscopy ... 581
Optical rotations ... 601
Order-disorder theory ... 538
Photosynthesis ... 151
Solid state symposium ... 224
Solvation and association ... 489
Transference experiment ... 516
Viscosity of mixtures ... 564
Pioneer summer institute: The G.E. Science Fellowship Program at Union College. E. K. Bacon ... 39
Plastic mounts for radioactive samples, Disposable. T. E. Burgess, et al. ... 208
Plating from an iodide complex, Silver. Tested demonstration ... A419
Plugboard teaching aid: Analog computer instruction. James O. Osburn ... 492
Polystyrene—a multistep synthesis. Samuel H. Wilen, Chester B. Kremer, and Irving Waltcher ... 304
Potentials, Electrode, with a vacuum tube voltmeter. Tested demonstration ... A637
Practical examination, laboratory, in quantitative chemistry. William M. MacNevin ... 144
Precipitation and solubility effects, A simple demonstration of some. E. Matijevic, J. P. Kratohvil, and M. Kerker ... 307
Precipitation from homogeneous solution: A lecture demonstration. Louis Gordon and Eugene D. Salesin ... 16
Precipitation, Heat of: A general chemistry experiment. H. Lawrence Clever ... 470
Preparation of metallic lithium. Tested demonstration ... A419
Preparative separations replace typical qualitative analysis. Carl Mahr ... 407
Radiation detectors, Nuclear. Chemical instrumentation. S. Z. Lewin ... A135
Radiation, Nuclear, electronic gear. Chemical instrumentation. S. Z. Lewin ... A225, A305, A389, A457
Radioactive samples, Disposable plastic mounts for. T. E. Burgess, et al. ... 208
Radioactivity, extinct natural, Chronology of nucleosynthesis and. Truman P. Kohman ... 73
Radioactivity sources, Convenient. D. J. Carswell ... 352
Radiochemical experiment, Ion exchange. J. F. Mucci, et al. ... 348
Radiochemistry
Chemical instrumentation ... A225
Half life of UX_2 (Pa^{234}) ... 31
Resolving time ... 29, (L)329
Techniques ... 344
Radioisotope demonstration of common ion effect on solubility. E. Roig, I. G. Rieckehoff, C. S. Russo, and J. D. Curet ... 330
Radioisotope techniques into the chemistry curriculum, Incorporating. Norman S. Radin ... 344
Raoult's law, A qualitative demonstration of. Tested demonstration ... A545
Rapid detection of cations of copper group. Ramesh Chandra ... 413
Rapid, direct-reading micro-balance. A. Daniel L. Harris ... 409
Rapid efficient method for reducing certain metallic isotopic oxides. Philip S. Baker ... 117
Rapid filtering without using a filter pump. Tested demonstration ... A813
Rates of consecutive reactions, Paper electrophoretic study of: The deamidation of vitamin B12. Grant H. Barlow ... 37

Terminology
Molar ... 549, 553, 555
Ternary diagrams. Textbook errors, 35. Karol J. Mysels ... 627
Tested demonstrations. Frederic B. Dutton, *Editor.*
Cis-trans isomerization ... A179
Combining volumes: Hydrogen chloride and ammonia ... A813
Condensation of noxious gases ... A357
Dalton's law of partial pressures ... A545
A demonstration answer to a stereochemical puzzler ... A289
Demonstration notes: Burning phosphorus in air, catalytic oxidation of ammonia, color change with temperature, sublimation ... A23
Demonstration notes: Common ion effect; chlorine water substitute; rapid filtering without using a filter pump; writing on beakers, flasks, etc.; boiling liquid freezes water ... A813
Displacement reactions—spongy tin ... A637
The effect of temperature on solubility ... A99
Electrode potentials with a vacuum tube voltmeter ... A637
Fire under water ... A751
Graham's law of diffusion using overhead projector ... A23
Introduction to Le Chatelier's principle ... A491
Photochemical catalysis of an aliphatic substitution reaction ... A289
Preparation of metallic lithium ... A419
Qualitative demonstration of Raoult's law ... A545
Reaction to introduce Lewis acid-base chemistry, A ... A491
Silver plating from an iodide complex ... A419
Surface tension ... A99
Synthesis of dimethylmaleate ... A179
Transition temperature in iron ... A357
"Visible" monomolecular films: Estima-
Texts, general chemistry, Coordination chemistry in. Roy W. Clark and Joel Selbin ... 466
Theories on the origin of meteorites. Edward Anders and Gordon G. Goles ... 58
There are electrons in atoms! Anatol Sementsov ... 22
Thermoanalytical kinetic experiment: The catalyzed reaction of phenyl isocyanate with butanol. L. B. Weisfeld ... 88
Thermobalance, A controlled atmosphere. Wesley W. Wendlandt ... 566
Thermobalance for student experiments. Rudolph S. Bottei and Paul L. Gerace ... 568
Thermodynamic properties, Total and static. Edward A. Fletcher ... 257
Thermodynamics
Bond energies ... 292
Diamond synthesis ... 484
Entropy ... 334
Gas dynamics ... 257
Gibbs-Duhem equation ... 509
Imperfect gases ... 140
Le Chatelier's principle ... 375
Order-disorder theory ... 538
Solid state ... 317
Three-dimensional representation of electrodeposition. W. H. Reinmuth ... 149
Tin(II), Benzenoid nitro-compounds as organic reagents for. Robert L. Burns and Fred M. Gruen ... 410
Tin, spongy—Displacement reactions. Tested demonstration ... A637
Titration, A conductometric. Analysis of aspirin. James S. Proctor and John E. Roberts ... 471
Titration controller for demonstrating the behavior of acid-base indicators. John T. Stock ... 633
Titrations with externally generated rea-
Turbidimetric determination of lead, The. Gerald Perkins, Jr., J. W. Wimberley, J. F. Lamb, and L. E. Maurer ... 358

Ultra low conductivity water. National Bureau of Standards Summary Technical Report ... 421
Undergraduate training of chemistry majors: A report of the Bucknell Conference. Bennett R. Willeford, Jr., and Leallyn B. Clapp ... 251
Union College, The G.E. Science Fellowship Program at. A pioneer summer institute. E. K. Bacon ... 39
Uses and abuses of bond energies. Bruce E. Knox and Howard B. Palmer ... 292
Using filmed demonstrations in general chemistry. Ruth Botdorf ... 95
Using the decay of Ba-137 to determine resolving time in G-M counting. Robert L. Stearns and Joseph F. Mucci ... 29, (L)329

Vacuum diffusion pumps, Safety device for. G. O. Pritchard and G. H. Miller ... 525
Vapor pressure apparatus, Isopiestic. G. W. Dailey, et al. ... 28
Variable transformer, A motor-driven. Meyer M. Markowitz, et al. ... 96
Vibrating molecular models: Frequency shifts in strained ring double bonds. Norman B. Colthup ... 394
Viscosities of butanol-heptane mixtures. K. Keith Innes ... 564
"Visible" monomolecular films: Estimation of molecular size and Avogadro's number. Tested demonstration ... A751
Vitalism, vitality without. The Liebig-

Ratio errors in pseudo first order reactions. Fred Sicilio and M. D. Peterson... 576
Reaction kinetics by differential thermal analysis. Wesley W. Wendlandt... 571
Reaction to introduce Lewis acid-base chemistry. Tested demonstration..... A491
Reactive intermediates of organic chemistry. Ross Stewart..... 308
Recent advances in inorganic chemistry. Symposium. Introduction. R. W. Parry..... 160
Recent advances in solid state inorganic materials. Otto L. Forchheimer and Terry F. Newkirk..... 183
Recent developments in metal carbonyl synthesis. Harold E. Podall..... 187
Recent developments in N—F chemistry. Charles B. Colburn..... 180
Recent developments in semiconductors. W. Crawford Dunlap..... 238
Recent improvements in our knowledge of cosmic abundances. William D. Ehmann..... 53
Redefinition of "Mole," A. Shiu Lee..... 549
Redetermination of the half-life of carbon-14. National Bureau of Standards Office of Technical Information..... 82
Redox revisited. Karl L. Lockwood .....326, (L)637
Reducing agents, New selective. Herbert C. Brown..... 173
Reducing certain metallic isotopic oxides, A rapid efficient method for. Philip S. Baker..... 117
Re-jargonizing the little-languages of science, Translating and. Jack De Ment..... 415
Research ideas for young chemists—*See Chemical projects*
Research participation program for high school students, A summer. Reed F. Riley and C. G. Overberger..... 424
Residue, Pesticide, control under the Food, Drug, and Cosmetic Act. Curtis R. Joiner..... 370
Resolving time in G-M counting, Using the decay of Ba-137 to determine. Robert L. Stearns and Joseph F. Mucci.....29, (L)329
Reviews of ACS Cooperative Exams..... 210
Revised teaching of biochemistry. Howard M. Myers..... 363
Rheology in the world of neglected dimensions. John D. Ferry..... 110
Ribonucleic acid and protein synthesis. Jay S. Roth..... 217
Richard Willstätter. Rolf Huisgen..... 10
Robert Maskell Patterson: First to teach atomic theory in America? Wyndham D. Miles..... 561
Role of chemistry in computers, The. A. H. Sporer..... 323
Rotation, optical, A model for. Leon L. Jones and Henry Eyring..... 601
Russian chemical literature, Abbreviations in. Julian F. Smith..... 41

S*afety*
Reaction shield..... 634
Test for acid wash..... 291
Safety device for vacuum diffusion pumps. G. O. Pritchard and G. H. Miller... 525
Salting-out chromatography: a review. William Rieman III..... 338
Saponification equivalents of phenolic esters, Determination of. S. W. Tobey, et al..... 611
"Sceptical Chymist," 1661. Roy G. Neville..... 106
Science Fair-International and the ACS Chemistry Awards, The National. Robert M. Brooker and Richard T. Rapala..... 209
Science fairs
Editorially speaking.....159, 277
Sciences and the humanities, The problem of the. Harold G. Cassidy..... 441
Semiconductors, Recent developments in. W. Crawford Dunlap..... 238
Separatory funnels, Substitute for. Marcel Jean..... 515
Shape of the 2p and related orbitals, The. Textbook errors, 28. Irwin Cohen... 20
Shield for small-scale apparatus, A convenient safety. Stanley H. Langer and Peter Rantages..... 634
Silver plating from an iodide complex. Tested demonstration..... A419
Simple demonstration of some precipitation and solubility effects. E. Matijevic, J. P. Kratohvil, and M. Kerker..... 397
Simple filtration technique for gravimetric determinations. Shirley W. Gaddis. 5
Simple method for cleaning buret tips, A. William Michels..... 548
Simple method for finding slopes. James P. Hoare..... 570
Simple test for nitric acid fumes. Alfred Chan, Jr..... 412
Simple test for the detection of phosphorus in organic compounds. Roger Ketcham and Anne de G. Low-Beer..... 414
Simplified moving-boundary transference cell. Stephen W. Tobey..... 516
Slopes, A simple method for finding. James P. Hoare..... 570
Solid sample identification analysis in high school chemistry. William Vasilakes..... 146
Solid state, Chemistry of the. Symposium. 224
Solid state, Electrochemistry of the. Allen B. Scott..... 317
Solid state inorganic materials, Recent advances in. Otto L. Forchheimer and Terry F. Newkirk..... 183
Solids and the gaseous corrosion of metals, Diffusion in. Walter J. Moore..... 232
Solubilities, molar, calculating from equilibrium constants. James N. Butler.. 460
Solubility effects, A simple demonstration of some precipitation and. E. Matijevic, et al..... 397
Solubility, Radioisotope demonstration of common ion effect on. E. Roig, et al. 350
Solubility, The effect of temperature on. Tested demonstration..... A99
Solutions, binary, alloys, gas adsorption, and ferromagnetism, Applications to. Elements of order-disorder theory. J. M. Honig..... 538
Solutions, Float method for the analysis of. James O. Osburn and Il C. Choi..... 578
Solutions, metal-in-amine, The nature of. E. Charles Evers..... 590
Solutions, volumetric, Apparatus for oxygen-sensitive. E. L. Martin and K. E. Bentley..... 264
Solvation and association from colligative properties by graphical means. George B. Smith, C. A. Hollingsworth, and Darl H. McDaniel..... 489
Sources, Convenient radioactivity. D. J. Carswell..... 352
Specific qualitative reagent for copper. T. R. Williams and Frederick Burton..... 413
Spectrometers, Infrared. Chemical instrumentation.....A5, A71
Spectroscopy, Inorganic infrared. John R. Ferraro..... 201
Spectroscopy, Nuclear magnetic resonance. John D. Roberts..... 581
Spring, Walther (1848–1911). (P) Jean Timmermans (Ralph E. Oesper, *Translator*)..... 422
Static thermodynamic properties, Total and. Edward A. Fletcher..... 257
Stereoisomerism of carbon compounds. William K. Noyce..... 23
Stirrers, A teflon bearing for. M. L. Kaplan, et al..... 577
Stoll, Arthur (P)..... 13
Strained ring double bonds, Frequency shifts in. Vibrating molecular models. Norman B. Colthup..... 394
Structural chemistry
Bonding..... 382
Dipole moments (TE)..... 522
Elementary course..... 530
Field theory..... 164
NMR spectroscopy..... 581
Rubber Stuart models..... 295
Stereoisomerism..... 23
Structure and spectra of nickel(II) and copper(II) complexes, The. W. Manch and W. Conard Fernelius... 192
Stuart-type molecular models, Inexpensive. William G. Hoover and Duward Shriver..... 295
Student experiment with filter paper electrophoresis. James E. Garvin..... 36
Sublimation. Tested demonstration..... A23
Substitute for separatory funnels. Marcel Jean..... 515
Substitution reactions of metal ions, Crystal field theory and. Ralph G. Pearson..... 164
Summer chemistry program for teenagers Joseph K. Obold and Thomas S. Briggs..... 47
Summer institute, A pioneer. The G.E. Science Fellowship Program at Union College. E. K. Bacon..... 39
Summer institutes: Review and future plans. William E. Morrell..... 448
Summer research participation program for high school students, A. Reed F. Riley and C. G. Overberger..... 424
Surface area of activated charcoal by Langmuir adsorption isotherm. Boleslaw Ludwik Dunicz..... 357
Surface chemistry
Monomolecular films demonstration..... A751
Surface tension. Tested demonstration.... A99
Swarts, Frederic (1866–1940). (P) Jean Timmermans (Ralph E. Oesper, *Translator*)..... 422
Swarts, Theodor (P)..... 120
Symmetrical organic molecules, Dipole moments of. Textbook errors, 34. Wilbur N. Moulton..... 522
Symposia
Chemistry of the solid state.....224, 312
Cosmochemistry..... 52
Recent advances in inorganic chemistry.. 160
Synthesis of diamond, The. H. Tracy Hall..... 484
Synthesis of dimethylmaleate. Tested demonstration..... A179
Systematic way to avoid Le Chatelier's principle in chemical reactions. Lewis Katz..... 375
Systematization of inorganic cyclic compounds. I. Haiduc..... 134

Taste tells! Floyd L. James..... 291
Teacher training
G. E. institute at Union College..... 39
NSF institutes.....448, 451
Teaching machines
Programs for chemistry 463
Teaching techniques
Analog computer..... 492
Biochemistry..... 363
Colored slides..... 473
Developing concepts..... 433
Examinations..... 478
General chemistry content..... 235
Geometric approach ..123, (L)329, (E)329, 509
Modern analysis..... 455
No Le Chatelier's principle..... 375
Programmed instruction..... 463
Redox survey.....326, (L)637
Using filmed demonstrations..... 95
Technical writing
Jargon..... 415
Teenagers, A summer chemistry program for. Joseph K. Obold and Thomas S. Briggs..... 47
Teflon bearing for stirrers. M. L. Kaplan, R. E. Crocker, and Herbert Sargent 577
Temperature and power measurements in precision solution calorimetry. William F. O'Hara, Ching-Hsien Wu, and Loren G. Hepler..... 512

Terminology
Molar 549, 553, 555
Ternary diagrams. Textbook errors, 35. KAROL J. MYSELS 627
Tested demonstrations. FREDERIC B. DUTTON, *Editor.*
Cis-trans isomerization A179
Combining volumes: Hydrogen chloride and ammonia A813
Condensation of noxious gases A357
Dalton's law of partial pressures A545
A demonstration answer to a stereochemical puzzler A289
Demonstration notes: Burning phosphorus in air, catalytic oxidation of ammonia, color change with temperature, sublimation A23
Demonstration notes: Common ion effect; chlorine water substitute; rapid filtering without using a filter pump; writing on beakers, flasks, etc.; boiling liquid freezes water A813
Displacement reactions—spongy tin A637
The effect of temperature on solubility ... A99
Electrode potentials with a vacuum tube voltmeter A637
Fire under water A751
Graham's law of diffusion using overhead projector A23
Introduction to Le Chatelier's principle ... A491
Photochemical catalysis of an aliphatic substitution reaction A289
Preparation of metallic lithium A419
Qualitative demonstration of Raoult's law A545
Reaction to introduce Lewis acid-base chemistry, A A491
Silver plating from an iodide complex A419
Surface tension A99
Synthesis of dimethylmaleate A179
Transition temperature in iron A357
"Visible" monomolecular films: Estimation of molecular size and Avogadro's number A751
Textbook errors. Karol J. Mysels, *Editor.*
28. The shape of the 2*p* and related orbitals. IRWIN COHEN 20
29. The existence of endothermic adsorption. JOHN M. THOMAS 138
30. The ionization constant of water. THEDFORD P. DIRKSE 260
31. The pyrolytic decomposition of carboxylate salts to ketones. HARRY P. SCHULTZ AND JACKSON P. SICHELS 300
32. The equilibria of complex formation. JAMES E. BANKS 391
33. The paddle-wheel Crookes tube. J. ARTHUR CAMPBELL 480
34. Dipole moments of symmetrical organic molecules. WILBUR N. MOULTON 522
35. Miscellanea No. 4: Chromyl chloride test for chlorides; "monovalent oxygen" compounds; ternary diagrams. K. J. MYSELS 627
Texts, general chemistry, Coordination chemistry in. ROY W. CLARK AND JOEL SELBIN 466
Theories on the origin of meteorites. EDWARD ANDERS AND GORDON G. GOLES .. 58
There are electrons in atoms! ANATOL SEMENTSOV 22
Thermoanalytical kinetic experiment: The catalyzed reaction of phenyl isocyanate with butanol. L. B. WEISFELD 88
Thermobalance, A controlled atmosphere. WESLEY W. WENDLANDT 566
Thermobalance for student experiments. RUDOLPH S. BOTTEI AND PAUL L. GERACE 568
Thermodynamic properties, Total and static. EDWARD A. FLETCHER 257
Thermodynamics
Bond energies 292
Diamond synthesis 484
Entropy 334
Gas dynamics 257
Gibbs-Duhem equation 509
Imperfect gases 140
Le Chatelier's principle 375
Order-disorder theory 538
Solid state 317
Three-dimensional representation of electrodeposition. W. H. REINMUTH 149
Tin(II), Benzenoid nitro-compounds as organic reagents for. ROBERT L. BURNS AND FRED M. GRUEN 410
Tin, spongy—Displacement reactions. Tested demonstration A637
Titration, A conductometric. Analysis of aspirin. JAMES S. PROCTOR AND JOHN E. ROBERTS 471
Titration controller for demonstrating the behavior of acid-base indicators. JOHN T. STOCK 633
Titrations with externally generated reagents, An inexpensive coulometer for. WILLIAM F. HEAD, JR. AND MAX M. MARSH 361
Total and static thermodynamic properties: Old gas dynamic properties which may be new to the chemist. EDWARD A. FLETCHER 257
Toxic reagents, Device for charging microtubes with. JOHN T. STOCK ET AL. 524
Training, graduate, of eminent chemists, Institutional influences in the. B. R. SIEBRING 630
Transference cell, Simplified moving-boundary. STEPHEN W. TOBEY 516
Transformer, A motor-driven variable. MEYER M. MARKOWITZ, ET AL. 96
Transition temperature in iron. Tested demonstration A357
Transulating and re-jargonizing the little-languages of science. JACK DE MENT .. 415
Treasurer's report, Division of Chemical Education, ACS. WILLIAM G. KESSEL 635
Turbidimetric determination of lead, The. GERALD PERKINS, JR., J. W. WIMBERLEY, J. F. LAMB, AND L. E. MAURER 358
ULTRA low conductivity water. National Bureau of Standards Summary Technical Report 421
Undergraduate training of chemistry majors: A report of the Bucknell Conference. BENNETT R. WILLEFORD, JR., AND LEALLYN B. CLAPP 251
Union College, The G.E. Science Fellowship Program at. A pioneer summer institute. E. K. BACON 39
Uses and abuses of bond energies. BRUCE E. KNOX AND HOWARD B. PALMER 292
Using filmed demonstrations in general chemistry. RUTH BOTDORF 95
Using the decay of Ba-137 to determine resolving time in G-M counting. ROBERT L. STEARNS AND JOSEPH F. MUCCI ..... 29, (L)329
VACUUM diffusion pumps, Safety device for. G. O. PRITCHARD AND G. H. MILLER ... 525
Vapor pressure apparatus, Isopiestic. G. W. DAILEY, ET AL. 28
Variable transformer, A motor-driven. MEYER M. MARKOWITZ, ET AL. 96
Vibrating molecular models: Frequency shifts in strained ring double bonds. NORMAN B. COLTHUP 394
Viscosities of butanol-heptane mixtures. K. KEITH INNES 564
"Visible" monomolecular films: Estimation of molecular size and Avogadro's number. Tested demonstration A751
Vitalism, vitality without. The Liebig-Pasteur controversy. GEORGE E. HEIN 614
Vitamin B_{12}, the deamidation of: Paper electrophoretic study of rates of consecutive reactions. GRANT H. BARLOW 37
Volumes, Combining: Hydrogen chloride and ammonia. Tested demonstration. A813
WALTHER Spring (1848–1911). (P) JEAN TIMMERMANS (RALPH E. OESPER, *Translator*) 422
Water by electrodialysis, Deionized. J. A. SCHUFLE 17
Water, The ionization constant of. Textbook errors. 30. THEDFORD P. DIRKSE 260
Water, Ultra low conductivity. National Bureau of Standards Summary Technical Report 421
Wieland, Heinrich (P) 15
Willstätter, Richard. (P) ROLF HUISGEN .. 10
Writing on beakers, flasks, etc. Tested demonstration A813

BOOKS REVIEWED

TITLES

ABOUT chemistry. MAGNUS PYKE 379
Abundance of chemical elements. V. V. CHERDYNTSEV 640
Advanced organic chemistry. G. W. WHELAND 221
Advances in fluorine chemistry. Vol. 1. M. STACEY, J. C. TATLOW, AND A. G. SHARPE, *Editors* A270
Advances in organic chemistry: Methods and results. Vols. 1 and 2. RALPH A. RAPHAEL, E. C. TAYLOR, AND HANS WYNBERG, *Editors* A438
Advances in protein chemistry. Vol. 14. C. B. ANFINSEN, JR., ET AL., *Editors* .. A54
AEDT y similares, Aplicaciones analíticas del. FRANCISCO BERMEJO MARTINEZ AND ANTONIO PRIETO BOUZA 274
Agricultural chemists, Official methods of analysis of. WILLIAM HORWITZ, *Editor.* 431
Alchemie zur Grossynthese, Von der. Grosse Chemiker. RUDOLF SACHTLEBEN AND ARMIN HERMANN 380
American Chemical Society specifications, 1960. Reagent chemicals. 1960 ACS Committee on Analytical Reagents ... A855
Amino acids, proteins, and cancer biochemistry. JOHN T. EDSALL, *Editor* .. A428
Ammonia, manufacture and uses. A. J. HARDING A196
Analysis, Chemical. Vol. 12: Systematic analysis of surface-active agents. MILTON J. ROSEN AND HENRY A. GOLDSMITH A52
Analysis, chemical, Physical methods in. Vol. 1. 2nd rev. ed. WALTER G. BERL, *Editor* 104
Analysis, inorganic, The use of organic reagents in. A. I. BUSEV AND N. G. POLIANSKII A922
Analysis of air pollutants, The chemical. MORRIS B. JACOBS A114

Analysis, Organic. Vol. 4. JOHN MITCHELL, JR., I. M. KOLTHOFF, E. S. PROSKAUER, AND A. WEISSBERGER, *Editors* A638
Analysis, Quantitative inorganic. R. BELCHER A44
Analytical chemical separations, Qualitative analysis and. PHILIP W. WEST AND MAURICE M. VICK A642
Analytical chemistry, Calculations of. LEICESTER F. HAMILTON AND STEPHEN G. SIMPSON 49
Analytical chemistry, Comprehensive. Vol. 1B, Classical analysis. CECIL L. WILSON AND DAVID W. WILSON A766
Analytical chemistry of the rare earths. R. C. VICKERY A768
Analytical chemistry, Treatise on. I. M. KOLTHOFF AND PHILIP J. ELVING. Part 1: Theory and Practice. Vol. 2. .... A842
Part 2: Analytical chemistry of the elements. Vol. 1 ..... A844
Analytical methods, mechanisms and. Toxicology. C. P. STEWART AND A. STOLMAN, *Editors* A923
Anorganische Chemie. Band 1. ISTVAN NARAY-SZABO 380
Anorganischen chemie, Gmelins handbuch der. 8th ed. E. H. B. PIETSCH, *Editor*
System 15: Silicon, part C A432
System 33: Cadmium supplement A372
Anorganisches grundpraktikum. CARL MAHR A916
Aplicaciones analíticas del AEDT y similares. FRANCISCO B. MARTINEZ AND ANTONIO P. BOUZA 274
Atom, The book of the. LEONARD DE VRIES, *translated by* ERIC G. BREEZE .. 430
Atoms, molecules and chemical change. ERNEST GRUNWALD AND RUSSELL H. JOHNSEN 49
Atoms to stars, From. THEODORE A. ASHFORD 103

BIOCHEMISTRY, Comparative: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors*. Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
Biochemistry, Comparative, of photoreactive systems. MARY BELLE ALLEN, *Editor* 432
Biochemistry. Experimental. GERALD LITWACK 221
Biochemistry of plants and animals. M. FRANK MALLETTE, PAUL M. ALTHOUSE, AND CARL O. CLAGETT A282
Biochemistry, Radioactive isotopes in. ENGELBERT BRODA 536
Biology, Introduction to a submolecular. ALBERT SZENT-GYÖRGYI 276
Book of the atom, The. LEONARD DE VRIES, *translated by* ERIC G. BREEZE .. 430

CADMIUM supplement, System 33. Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. PIETSCH, *Editor* A372
Calculations of analytical chemistry. LEICESTER F. HAMILTON AND STEPHEN G. SIMPSON 49
Cancer biochemistry, Amino acids, proteins, and. JOHN T. EDSALL, *Editor* A428
Careers and opportunities in science. PHILIP POLLACK A636
Catalysis, heterogeneous, The mechanism of. J. H. DE BOER, *Editor* A494
Cavendish, Henry: His life and scientific work. A. J. BERRY A40
Ceramics, Introduction to. W. D. KINGERY A548
Chemical analysis. Vol. 12: Systematic analysis of surface-active agents. MILTON J. ROSEN AND HENRY A. GOLDSMITH A52
Chemical analysis of air pollutants, The. MORRIS B. JACOBS A114
Chemical change, Atoms, molecules, and. ERNEST GRUNWALD AND RUSSELL H. JOHNSEN 49
Chemical engineering practice. Vol. 10: Ancillary services. HERBERT W. CREMER AND SIDNEY B. WATKINS, *Editors*. 585
Chemical periodicity. R. T. SANDERSON .. 274
Chemical properties and identification of ions: An introduction to semi-micro qualitative analysis. OMER K. WHIPPLE A920
Chemical senses in health and disease, The. H. KALMUS AND S. J. HUBBARD A112
Chemical technology, Encyclopedia of (Edited by RAYMOND E. KIRK AND DONALD F. OTHMER). Second supplement volume. ANTHONY STANDEN, *Editor* A370
Chemistry. MICHELL J. SIENKO AND ROBERT A. PLANE 585
Chemistry for engineers. EDWARD CARTMELL 222
Chemistry of carbon compounds. Vol. 4. Part C: Heterocyclic compounds. E. H. RODD, *Editor* 588
Chemistry of lipids in health and disease. The. H. K. KING A284
Chemistry of nucleic acids, The. D. O. JORDAN 586
Chemistry of yttrium and scandium, The. R. C. VICKERY A268
Chimie minérale, Nouveau traité de. PAUL PASCAL, *Editor*. Vol. 7 ..... A116
Vol. 15 331
Clinical chemistry, Standard methods of. Vol. 3. DAVID SELIGSON, *Editor* 639
Cobalt: Its chemistry, metallurgy and uses. ROLAND S. YOUNG, *Editor* A498
College chemistry: A systematic approach. HARRY H. SISLER, CALVIN A. VANDERWERF, AND ARTHUR W. DAVIDSON ... A554
Colloid chemistry, Introduction to. KAROL J. MYSELS 50
Comparative biochemistry: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors*. Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
Comparative biochemistry of photoreactive systems. MARY BELLE ALLEN, *Editor* 432
Complex compounds, Instability constants of. K. B. YATSIMIRSKII AND V. P. VASIL'EV 157
Comprehensive analytical chemistry. Vol. 1B, Classical analysis. CECIL L. WILSON AND DAVID W. WILSON A766
Conservation, Teaching science through. MARTHA E. MUNZER AND PAUL F. BRANDWEIN 275
Crystal-structure analysis. M. J. BUERGER A364
Crystal structures. Supplement 5. W. G. WYCKOFF A770
Crystals and the polarizing microscope. N. H. HARTSHORNE AND A. STUART ... A121

DETONATION, Theory of. IA. B. ZELDOVICH AND A. S. KOMPANEETS 330
Dispensatory of the United States of America, The. New drug developments volume. ARTHUR OSOL, *Editor*. A926
Dispersion, Optical rotatory: Applications to organic chemistry. CARL DJERASSI. A48
Distillation, Molecular. G. BURROWS 536

EDUCATION, Policies, for science. FREDERICK L. FITZPATRICK, *Editor* 331
Einstein: A pictorial biography, WILLIAM CAHN 275
Electrolytic manufacture of chemicals from salt. D. W. F. HARDIE A196
Electronics for spectroscopists. C. G. CANNON, *Editor* A918
Electrophoresis in physiology. LENA A. LEWIS AND M. D. POULIK A52
Electrophoresis, The principles of. RENE AUDUBERT AND SERGE DE MENDE ... A52
Elements, Abundance of chemical. V V. CHERDYNTSEV 640
Elements, Analytical chemistry of the. Vol. 1 of Part 2 of Treatise on analytical chemistry. I. M. KOLTHOFF AND PHILIP J. ELVING A844
Encyclopedia of chemical technology (*Edited by* R. E. KIRK AND D. F. OTHMER). Second supplement vol. ANTHONY STANDEN, *Editor* A370
Engineering thermodynamics: An introductory textbook. JAMES B. JONES AND GEORGE A. HAWKINS A50
Engineers, Chemistry for. EDWARD CARTMELL 222
Experimental biochemistry. GERALD LITWACK 221
Experiments, chemistry, The golden book of. ROBERT BRENT A852
Experiments for instrumental methods. CHARLES N. REILLEY AND DONALD T. SAWYER 481
Experiments, Radioisotope, for the chemistry curriculum. NORMAN RADIN, *Editor* 379
FATTY acids: Their chemistry, properties, production, and uses. Part 1. KLARE S. MARKLEY, *Editor* A852
Fertility, The soil and its. H. TEUBCHER, R. ADLER, AND JEROME P. SEATON A286
Fluorine chemistry, Advances in. Vol. 1. M. STACEY, J. C. TATLOW, AND A. G. SHARPE, *Editors* A270
Food chemistry. LILLIAN HOAGLAND MEYER A202
Food processing, Nutritional evaluation of. ROBERT S. HARRIS AND HARRY VON LOESECKE, *Editors* A368
Free energy and biological function. Vol. 2 of Comparative biochemistry: A comprehensive treatise. MARCEL FLORKIN AND HOWARD S. MASON, *Editors* 158
From atoms to stars. THEODORE A. ASHFORD 103
Fuel cells. G. J. YOUNG, *Editor* 157

GENERAL chemistry. GARTH L. LEE AND HARRIS O. VAN ORDEN A110
General organic chemistry. WILLIAM C. STICKLER 534
Genetics, An outline of chemical. BERNARD S. STRAUSS A196
Geochemistry, Methods in. A. A. SMALES AND L. R. WAGNER, *Editors* A270
Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. PIETSCH, *Editor*.
System 15: Silicon, part C A432
System 33: Cadmium supplement A372
Golden book of chemistry experiments, The. ROBERT BRENT A852
Greek fire and gunpowder, A history of. J. R. PARTINGTON 430

HALOGENVERBINDUNGEN. Vol. 5, Part 4 of Methoden der organischen chemie. EUGENE MÜLLER, *Editor* A194
The Harvey Lectures. Series 54 222
Heat and thermodynamics. J. K. ROBERTS. 158
Henry Cavendish: His life and scientific work. A. J. BERRY A40
Heterocyclic compounds, An introduction to the chemistry of. R. M. ACHESON ... A430
Heterocyclic compounds, Part C of Vol. 4 of Chemistry of carbon compounds. E. H. RODD, *Editor* 588
History of chemistry, A short. J. R. PARTINGTON A374
History of Greek fire and gunpowder, A. J. R. PARTINGTON 430
History of metallography, A. CYRIL STANLEY SMITH 482

INORGANIC analysis, Quantitative. R. BELCHER A44

Inorganic chemistry: A guide to advanced study. R. B. Heslop and P. L. Robinson 331
Inorganic chemistry, Modern aspects of. H. J. Emeléus and J. S. Anderson ... 104
Inorganic chemistry, Progress in. Vol. 2. F. Albert Cotton, *Editor* A924
Inorganic chemistry, Synthetic. William L. Jolly 157
Inorganic chemistry, Textbook of. S. Young Tyree, Jr. and Kerro Knox. 481
Inorganic syntheses. Vol. 6. Eugene G. Rochow, *Editor* A552
Insecticides, Modern, and world food production. F. A. Gunther and L. R. Jeppson A634
Instability constants of complex compounds. K. B. Yatsimirskii and V. P. Vasil'ev. 157
Instrumental methods, Experiments for. Charles N. Reilley and Donald T. Sawyer 481
Introduction a l'etude des parfums. T. Bassiri A550
Introduction to a submolecular biology. Albert Szent-Györgyi 276
Introduction to ceramics. W. D. Kingery. A548
Introduction to colloid chemistry. Karol J. Mysels 50
Introduction to rubber technology. Maurice Morton, *Editor* 104
Introduction to solids. Leonid V. Azaroff. 586
Introduction to the chemistry of heterocyclic compounds. R. M. Acheson .. A430
Isotope effects on reaction rates. Lars Melander 330
Isotopes, Radioactive, in biochemistry. Engelbert Broda 536

Kinetics and mechanism. A. A. Frost and R. G. Pearson 535

Laboratory management and techniques. J. A. Edwards 587
Laboratory technique in organic chemistry. Kenneth B. Wiberg A192
Life, Viruses and the nature of. Wendell M. Stanley and Evans G. Valens ... 535
Lipids in health and disease, The chemistry of. H. K. King A284
Low-temperature techniques. F. Din and A. H. Cockett 275

Mechanism, Kinetics and. A. A. Frost and R. G. Pearson 535
Mechanism of heterogeneous catalysis, The. J. H. de Boer, *Editor* A494
Medicinal chemistry. Alfred Burger, *Editor* 276
Mental drugs: Chemistry's challenge to psychotherapy. O. A. Battista A550
Metallography, A history of. Cyril Stanley Smith 482
Metals and semiconductors, The surface chemistry of. Harry C. Gatos, *Editor* A548
Methoden der organischen chemie. Vol. 5, Part 4. Halogenverbindungen. Eugene Müller, *Editor* A194
Methods in geochemistry. A. A. Smales and L. R. Wagner, *Editors* A270
Microanalysis by the ring oven technique. Herbert Weiss 586
Microchemistry, Proceedings of the international symposium in A118
Micro-organisms, Polysaccharides of. M. Stacey and S. A. Barker 332
Modern aspects of inorganic chemistry. H. J. Emeléus and J. S. Anderson ... 104
Modern chemical processes. Vol. 6. Editors of *Industrial and Engineering Chemistry* A850
Modern insecticides and world food production. F. A. Gunther and L. R. Jeppson A634
Molecular distillation. G. Burrows 536
Molecular structure: The physical approach. J. C. D. Brand and J. C. Speakman 50
Molecules, and chemical change, Atoms. Ernest Grunwald and Russell H. Johnsen 49
Monographs for Teachers. Royal Institute of Chemistry, London, England 537

Name index of organic reactions. J. E. Gowan and T. S. Wheeler 588
Nouveau traité de chimie minérale. Paul Pascal, *Editor*. Vol. 7 ..... A116
Vol. 15 331
Nucleic acid outlines. Vol. 1. Van R. Potter A432
Nucleic acids, The. Vol. 3. Erwin Chargaff and J. N. Davidson, *Editors* A846
Nucleic acids, The chemistry of. D. O. Jordan 586
Nutritional evaluation of food processing. Robert S. Harris and Harry von Loesecke, *Editors* A368

Official methods of analysis of the Association of Official Agricultural Chemists. William Horwitz, *Editor* 431
Optical rotatory dispersion: Applications to organic chemistry. Carl Djerassi .. A48
Order, The search for. Cecil J. Schneer .. A108
Organic analysis. Vol. 4. John Mitchell, Jr., I. M. Kolthoff, E. S. Proskauer, and A. Weissberger, *Editors* A638
Organic analysis, Spot tests in. Fritz Feigl (*translated by* Ralph E. Oesper). A641
Organic chemistry. Allan R. Day and Madeleine M. Joullié 156
Organic chemistry. Keith M. Seymour .. 482
Organic chemistry, Advanced. G. W. Wheland 221
Organic chemistry, Advances in: Methods and results. Vols. 1 and 2. Ralph A. Raphael, E. C. Taylor, and Hans Wynberg, *Editors* A438
Organic chemistry, General. William C. Stickler 534
Organic chemistry, Laboratory technique in. Kenneth B. Wiberg A192
Organic chemistry, Physical methods of. Arnold Weissberger, *Editor* .. Part 2 222
Parts 3 and 4 432
Organic chemistry, Synthetic methods of. Vol. 14. W. Theilheimer 330
Organic chemistry, Tutorial questions in. P. A. Ongley A272
Organic compounds, Radiation chemistry of. A. J. Swallow 640
Organic reactions, Name index of. J. E. Gowan and T. S. Wheeler 588
Organic reactions. Vol. 11. Arthur C. Cope, *Editor-in-chief* 431
Organic reagents in inorganic analysis, The use of. A. I. Busev and N. G. Polianskii A923
Organic syntheses. Vol. 40. Melvin S. Newman, *Editor* A444
Organic systems, Oxidation-reduction potentials of. W. Mansfield Clark ... 158
Organischen chemie, Methoden der. Vol. 5, Part 4: Halogenverbindungen. Eugene Müller, *Editor* A194
Organometallic chemistry. H. Zeiss, *Editor* 587
Outline of chemical genetics. Bernard S. Strauss A196
Oxidation-reduction potentials of organic systems. W. Mansfield Clark 158

Parfums, Introduction a l'etude des. T. Bassiri A550
Periodicity, Chemical. R. T. Sanderson. 274
Pharmacy, Physical. Alfred N. Martin .. A121
Phosphorus esters, Toxic. Richard D. O'Brien 380
Photochemistry in the liquid and solid states. Lawrence J. Heidt, Robert S. Livingston, Eugene Rabinowitch, and Farrington Daniels, *Editors* ... A276
Photoreactive systems, Comparative biochemistry of. Mary Belle Allen, *Editor* 432
Physical approach, The: Molecular structure. J. C. D. Brand and J. C. Speakman 50
Physical chemistry. Farrington Daniels and Robert A. Alberty 639
Physical chemistry of surfaces. Arthur W. Adamson A366
Physical methods in chemical analysis. Vol. 1. 2nd rev. ed. Walter G. Berl, *Editor* 104
Physical methods of organic chemistry. Arnold Weissberger, *Editor*. Part 2. 222
Parts 3 and 4 432
Physical pharmacy. Alfred N. Martin .. A121
Physical science. Donald S. Allen and Richard J. Ordway 156
Physico-chemical constants of binary systems in concentrated solutions. Vol. 4: Systems with inorganic plus organic or inorganic compounds (excepting metallic derivatives). Jean Timmermans. A644
Physiology, Electrophoresis in. Lena A. Lewis and M. D. Poulik A52
Plastic, Source book of the new. Vol. 2. Herbert R. Simonds A855
Polarizing microscope, Crystals and the. N. H. Hartshorne and A. Stuart ... A121
Policies for science education. Frederick L. Fitzpatrick, *Editor* 331
Pollutants, The chemical analysis of air. Morris B. Jacobs A114
Polymer chemistry, Preparative methods of. Wayne Sorenson and Tod W. Campbell A840
Polymers, Properties and structure of. A. V. Tobolsky A922
Polysaccharides of micro-organisms. M. Stacey and S. A. Barker 332
Preparative methods of polymer chemistry. Wayne Sorenson and Tod W. Campbell A840
Principles of chemical thermodynamics. Charles E. Reid 50
Principles of electrophoresis, The. Rene Audubert and Serge de Mende A52
Proceedings of the International Symposium in Microchemistry A118
Process of education, The. Jerome S. Bruner A276
Processes, Modern chemical. Vol. 6. Editors of *Industrial and Engineering Chemistry* A850
Programmed learning, Teaching machines and. A. A. Lumsdaine and Robert Glaser, *Editors* 275
Progress in inorganic chemistry. Vol. 2. F. Albert Cotton, *Editor* A924
Properties and structure of polymers. A. V. Tobolsky A922
Protein chemistry, Advances. Vol. 14. C. B. Anfinsen, Jr., et al., *Editors* A54
Proteins, and cancer biochemistry, Amino acids. John T. Edsall, *Editor* A428
Psychotherapy, chemistry's challenge to. Mental drugs. O. A. Battista A550

Qualitative analysis, an introduction to semi-micro: Chemical properties and identification of ions. Omer K. Whipple A920
Qualitative analysis and analytical chemical separations. Philip W. West and Maurice M. Vick A642
Quantitative inorganic analysis. R. Belcher A44
Quantum chemistry: Methods and applications. R. Daudel, R. Lefebvre, and C. Moser 103

Radiation chemistry of organic compounds. A. J. Swallow 640
Radioactive isotopes in biochemistry. Engelbert Broda 536

Radiochemistry of the rare earths, scandium, yttrium and actinium, The. P. C. Stevenson and W. E. Nervik..... A768
Radioisotope experiments for the chemistry curriculum. Norman Radin, *Editor*.. 379
Radioisotope techniques. Ralph T. Overman and Herbert M. Clark..... A436
Rare earths, Analytical chemistry of the. R. C. Vickery..... A768
Rare earths, scandium, yttrium, and actinium, The radiochemistry of the. P. C. Stevenson and W. E. Nervik.. A768
Rates, Isotope effects on reaction. Lars Melander..... 330
Reagent chemicals. ACS specifications, 1960. 1960 ACS Committee on Analytical Reagents..... A855
Restless atom, The. Alfred Romer. The Science study series. Vol. 12..... 588
Ring oven technique, Microanalysis by the. Herbert Weisz..... 586
Rubber technology, Introduction to. Maurice Morton, *Editor*..... 104
Russian, Scientific. James W. Perry..... 535
Russian for scientists. C. R. Buxton and H. Sheldon Jackson..... A500

Salt, Electrolytic manufacture of chemicals from. D. W. F. Hardie..... A196
Scandium, The chemistry of yttrium and. R. C. Vickery..... A268
Science education, Policies for. Frederick L. Fitzpatrick, *Editor*..... 331
Science study series. Vol. 12. The restless atom. Alfred Romer..... 588
Vol. 18. Water, the mirror of science. Kenneth S. Davis and John A. Day. 588
Scientific Russian. James W. Perry..... 535
Search for order, The. Cecil J. Schneer. A108
Selective toxicity. Adrien Albert..... 332
Semiconductors, The surface chemistry of metals and. Harry C. Gatos, *Editor*. A548
Semi-micro qualitative analysis, An introduction to: Chemical properties and identification of ions. Omer K. Whipple..... A920
Senses, The chemical, in health and disease. H. Kalmus and S. J. Hubbard..... A112
Separation processes in practice. Robert F. Chapman, *Editor*..... 482
Short history of chemistry. J. R. Partington..... A374
Silicon, Part C, System 15. Gmelins handbuch der anorganischen chemie. 8th ed. E. H. E. Pietsch, *Editor*..... A432
Soil and its fertility, The. H. Teuscher, R. Adler and Jerome P. Seaton.... A286
Solids, Introduction to. Leonid V. Azároff..... 586
Source book of the new plastic. Vol. 2. Herbert R. Simonds..... A855
Spectrochemical analysis. L. H. Ahrens and S. R. Taylor..... A644
Spot tests in organic analysis. Fritz Feigl (*translated by* Ralph E. Oesper)..... A641
Standard methods of clinical chemistry. Vol. 3. David Seligson, *Editor*..... 639
Statistical manual for chemists. Edward L. Bauer..... A46
Statistical theory and methodology in science and engineering. K. A. Brownlee..... A370
Story of chemistry, The. Georg Lockemann..... A116
Structure of science, A. Joseph H. Simons..... 276
Style guide for chemists. Louis F. Fieser and Mary Fieser..... A440
Substitution at elements other than carbon. C. K. Ingold..... A434
Surface-active agents, Systematic analysis of. Chemical analysis. Vol. 12. Milton J. Rosen and Henry A. Goldsmith..... A52
Surface chemistry of metals and semiconductors, The. Harry C. Gatos, *Editor*..... A548
Surfaces, Physical chemistry of. Arthur W. Adamson..... A366
Syntheses, Inorganic. Vol. 6. Eugene G. Rochow, *Editor*..... A552
Syntheses, Organic. Vol. 40. Melvin S. Newman, *Editor*..... A444
Synthetic inorganic chemistry. William L. Jolly..... 157
Synthetic methods of organic chemistry. Vol. 14. W. Theilheimer..... 330

Tables of thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam. Joseph Hilsenrath, et al..... 536
Teaching machines and programmed learning. A. A. Lumsdaine and Robert Glaser, *Editors*..... 275
Teaching science through conservation. Martha E. Munzer and Paul F. Brandwein..... 275
Ternary systems. G. Masing (B. A. Rogers, *Translator*)..... A848
Textbook of chemistry. Stella Goostray and J. Rae Schwenck..... A766
Textbook of inorganic chemistry. S. Young Tyree, Jr. and Kerro Knox. 481
Theorie fondamentale du system periodique des elements. Stanislas Dockx..... 222
Theory of detonation. Ia. B. Zeldovich and A. S. Kompaneets..... 330
Thermal engineering. Harry L. Solberg, Orville C. Cromer, and Albert R. Spalding..... A204
Thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam, Tables of. Joseph Hilsenrath, et al..... 536
Thermodynamics, Engineering: An introductory textbook. James B. Jones and George A. Hawkins..... A50
Thermodynamics, Heat and. J. K. Roberts..... 158
Thermodynamics, Principles of chemical. Charles E. Reid..... 50
Thermoelectricity. Paul H. Egli, *Editor*. 534
Toxic phosphorus esters. Richard D. O'Brien..... 380
Toxicity, Selective. Adrien Albert..... 332
Toxicology: Mechanisms and analytical methods. C. P. Stewart and A. Stolman, *Editors*..... A923
Transport phenomena. R. Byron Bird, Warren E. Stewart, and Edwin N. Lightfoot..... A640
Transport and thermodynamic properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam, Tables of. Joseph Hilsenrath, et al..... 536
Treatise on analytical chemistry. I. M. Kolthoff and Philip J. Elving. Part 1: Theory and practice. Vol. 2.. A842
Part 2: Analytical chemistry of the elements. Vol. 1..... A844
Tutorial questions in organic chemistry. P. A. Ongley..... A272

Ultrafiltration. L. Ambard and S. Trautmann..... 588
Use of organic reagents in inorganic analysis. A. I. Busev and N. G. Polianskii... A923

Valency, Wave mechanics and. J. W. Linnett..... 431
Viruses and the nature of life. Wendell M. Stanley and Evans G. Valens... 535
Von der Alchemie zur Grossynthese. Grosse Chemiker. Rudolf Sachtleben and Armin Hermann..... 380

Water, the mirror of science. Kenneth S. Davis and John Arthur Day. The Science study series. Vol. 18..... 588
Wave mechanics and valency. J. W. Linnett..... 431

X-ray absorption and emission in analytical chemistry. H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, and P. D. Zemany..... 430

Yttrium and scandium, The chemistry of. R. C. Vickery..... A268

AUTHORS

Acheson, R. M. An introduction to the chemistry of heterocyclic compounds.. A430
ACS, 1960 Committee on Analytical Reagents. Reagent chemicals. American Chemical Society specifications, 1960..... A855
Adamson, Arthur W. Physical chemistry of surfaces..... A366
Ahrens, L. H., and Taylor, S. R. Spectrochemical analysis..... A644
Albert, Adrien. Selective toxicity..... 332
Alberty, Robert A.—*See* Daniels, Farrington
Allen, Donald S., and Ordway, Richard J. Physical science..... 156
Allen, Mary Belle, *Editor*. Comparative biochemistry of photoreactive systems..... 432
Althouse, Paul M.—*See* Mallette, M. Frank
Ambard, L., and Trautmann, S. Ultrafiltration..... 588
Anfinsen, C. B., Jr., Anson, M. L., Bailey, Kenneth, and Edsall, John T., *Editors*. Advances in protein chemistry. Vol. 14..... A54
Anderson, J. S.—*See* Emeléus, H. J.
Anson, M. L.—*See* Anfinsen, C. B., Jr.
Ashford, Theodore A. From atoms to stars..... 103
Audubert, Rene and de Mende, Serge. The principles of electrophoresis..... A52
Azaroff, Leonid V. Introduction to solids..... 586

Bailey, Kenneth—*See* Anfinsen, C. B., Jr.
Barker, S. A.—*See* Stacey, M.
Bassiri, T. Introduction a l'etude des parfums..... A550
Battista, O. A. Mental drugs: Chemistry's challenge to psychotherapy.... A550
Bauer, Edward L. A statistical manual for chemists..... A46
Belcher, R. Quantitative inorganic analysis..... A44
Berl, Walter G., *Editor*. Physical methods in chemical analysis. Vol. 1. 2nd rev. ed..... 104
Berry, A. J. Henry Cavendish: His life and scientific work..... A40
Bird, R. Byron, Stewart, Warren E., and Lightfoot, Edwin N. Transport phenomena..... A640
Bouza, Antonio—*See* Martinez, Francisco
Brand, J. C. D. and Speakman, J. C. Molecular structure: The physical approach..... 50
Brandwein, Paul F.—*See* Munzer, Martha E.
Breese, Eric G., *Translator*. The book of the atom, by Leonard de Vries.
Brent, Robert. The golden book of chemistry experiments..... A852
Broda, Engelbert. Radioactive isotopes in biochemistry..... 536
Brownlee, K. A. Statistical theory and methodology in science and engineering..... A370
Bruner, Jerome S. The process of education..... A276
Buerger, M. J. Crystal-structure analysis..... A364

BURGER, ALFRED, *Editor.* Medicinal chemistry 276
BURROWS, G. Molecular distillation 536
BUSEV, A. I., AND POLIANSKII, N. G. The use of organic reagents in inorganic analysis A923
BUXTON, C. R., AND JACKSON, H. SHELDON. Russian for scientists A500

CAHN, WILLIAM. Einstein: A pictorial biography 275
CAMPBELL, TOD W.—*See* SORENSON, WAYNE
CANNON, C. G., *Editor.* Electronics for spectroscopists A918
CARTMELL, EDWARD. Chemistry for engineers 222
CHAPMAN, ROBERT F., *Editor.* Separation processes in practice 482
CHARGAFF, ERWIN, AND DAVIDSON, J. N., *Editors.* The nucleic acids.—Vol. 3 .. A846
CHERDYNTSEV, V. V. Abundance of chemical elements 640
CLAGETT, CARL O.—*See* MALLETTE, M. FRANK
CLARK, HERBERT M.—*See* OVERMAN, RALPH T.
CLARK, W. MANSFIELD. Oxidation-reduction potentials of organic systems. 158
COCKETT, A. H.—*See* DIN, F.
COPE, ARTHUR C., *Editor.* Organic reactions. Vol. 11 431
COTTON, F. ALBERT, *Editor.* Progress in inorganic chemistry. Vol. 2 ..... A924
CREMER, HERBERT W., AND WATKINS, SIDNEY B., *Editors.* Chemical engineering practice. Vol. 10: Ancillary services 585
CROMER, ORVILLE C.—*See* SOLBERG, HARRY L.

DANIELS, FARRINGTON, AND ALBERTY, ROBERT A. Physical chemistry 639
—*See* HEIDT, LAWRENCE J.
DAUDEL, R., LEFEBVRE, R. AND MOSER, C. Quantum chemistry: Methods and applications 103
DAVIDSON, ARTHUR W.—*See* SISLER, HARRY H.
DAVIDSON, J. N.—*See* CHARGAFF, ERWIN
DAVIS, KENNETH S., AND DAY, JOHN ARTHUR. Water, the mirror of science. Science study series. Vol. 18 588
DAY, ALLAN R. AND JOULLIÉ, MADELEINE M. Organic chemistry 156
DAY, JOHN ARTHUR—*See* DAVIS, KENNETH S.
DE BOER, J. H., *Editor.* The mechanism of heterogeneous catalysis A494
DE MENDE, SERGE—*See* AUDUBERT, RENE
DE VRIES, LEONARD. The book of the atom. (*Translated by* ERIC G. BREEZE) 430
DIN, F. AND COCKETT, A. H. Low-temperature techniques 275
DJERASSI, CARL. Optical rotatory dispersion: Applications to organic chemistry A48
DOCKX, STANISLAS. Theorie fondamentale du system periodique des elements 222

EDITORS of *Industrial and Engineering Chemistry.* Modern chemical processes. Vol. 6 ..... A850
EDSALL, JOHN T., *Editor.* Amino acids, proteins, and cancer biochemistry A428
—*See* ANFINSEN, C. B., JR
EDWARDS, J. A. Laboratory management and techniques 587
EGLI, PAUL H., *Editor.* Thermoelectricity. 534
ELVING, PHILIP J.—*See* KOLTHOFF, I. M.
EMELÉUS, H. J., AND ANDERSON, J. S. Modern aspects of inorganic chemistry. 104

FEIGL, FRITZ (*translated by* OESPER, RALPH E.). Spot tests in organic analysis ... A641
FIESER, LOUIS F., AND FIESER, MARY. Style guide for chemists A440
FIESER, MARY—*See* FIESER, LOUIS F.
FITZPATRICK, FREDERICK L., *Editor.* Policies for science education 331
FLORKIN, MARCEL, AND MASON, HOWARD S., *Editors.* Comparative biochemistry: A comprehensive treatise.
Vol. 1. Sources of free energy A442
Vol. 2. Free energy and biological function 158
FROST, A. A., AND PEARSON, R. G. Kinetics and mechanism 535

GATOS, HARRY C., *Editor.* The surface chemistry of metals and semiconductors A548
GLASER, ROBERT—*See* LUMSDAINE, A. A.
GOLDSMITH, HENRY A.—*See* ROSEN, MILTON J.
GOOSTRAY, STELLA, AND SCHWENCK, J. RAE. A textbook of chemistry A766
GOWAN, J. E., AND WHEELER, T. S. Name index of organic reactions 588
GRUNWALD, ERNEST, AND JOHNSEN, RUSSELL H. Atoms, molecules and chemical change 49
GUNTHER, F. A., AND JEPPSON, L. R. Modern insecticides and world food production A634

HAMILTON, LEICESTER F., AND SIMPSON, STEPHEN G. Calculations of analytical chemistry 49
HARDIE, D. W. F. Electrolytic manufacture of chemicals from salt A196
HARDING, A. J. Ammonia, manufacture and uses A196
HARRIS, ROBERT S., AND LOESECKE, HARRY VON, *Editors.* Nutritional evaluation of food processing A368
HARTSHORNE, N. H. AND STUART, A. Crystals and the polarizing microscope A121
HAWKINS, GEORGE A.—*See* JONES, JAMES B.
HEIDT, LAWRENCE J., LIVINGSTON, ROBERT S., RABINOWITCH, EUGENE, AND DANIELS, FARRINGTON, *Editors.* Photochemistry in the liquid and solid states A276
HERMANN, ARMIN—*See* SACHTLEBEN, RUDOLF
HESLOP, R. B. AND ROBINSON, P. L. Inorganic chemistry: A guide to advanced study 331
HILSENRATH, JOSEPH, ET AL. Tables of thermodynamic and transport properties of air, argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, oxygen, and steam 536
HORWITZ, WILLIAM, *Editor.* Official methods of analysis of the Association of Official Agricultural Chemists 431
HUBBARD, S. J.—*See* KALMUS, H.

INGOLD, C. K. Substitution at elements other than carbon A434

JACKSON, H. SHELDON—*See* BUXTON, C. R.
JACOBS, MORRIS B. The chemical analysis of air pollutants A114
JEPPSON, L. R.—*See* GUNTHER, F. A.
JOHNSEN, RUSSELL H.—*See* GRUNWALD, ERNEST
JOLLY, WILLIAM L. Synthetic inorganic chemistry 157
JONES, JAMES B. AND HAWKINS, GEORGE A. Engineering thermodynamics: An introductory textbook A50
JORDAN, D. O. The chemistry of nucleic acids 586
JOULLIÉ, MADELEINE M.—*See* DAY, ALLAN R.

KALMUS, H. AND HUBBARD, S. J. The chemical senses in health and disease .. A112
KING, H. K. The chemistry of lipids in health and disease A284
KINGERY, W. D. Introduction to ceramics. A548
KIRK, RAYMOND E. AND OTHMER, DONALD F. Encyclopedia of chemical technology. Second supplement volume, Edited by STANDEN, ANTHONY A370
KNOX, KERRO—*See* TYREE, S. YOUNG, JR.
KOLTHOFF, I. M., AND ELVING, PHILIP J. Treatise on analytical chemistry. Part 1: Theory and practice. Vol. 2 .. A842
Part 2: Analytical chemistry of the elements. Vol. 1 ..... A844
—*See* MITCHELL, JOHN, JR.
KOMPANEETS, A. S.—*See* ZELDOVICH, IA. B.

LEE, GARTH L., AND VAN ORDEN, HARRIS O. General chemistry A110
LEFEBVRE, R.—*See* DAUDEL, R.
LEWIS, LENA A., AND POULIK, M. D. Electrophoresis in physiology A52
LIEBHAFSKY, H. A., PFEIFFER, H. G., WINSLOW, E. H., AND ZEMANY, P. D. X-ray absorption and emission in analytical chemistry 430
LIGHTFOOT, EDWIN N.—*See* BIRD, R. BYRON
LINNETT, J. W. Wave mechanics and valency 431
LITWACK, GERALD. Experimental biochemistry 221
LIVINGSTON, ROBERT S.—*See* HEIDT, LAWRENCE J.
LOCKEMANN, GEORG. The story of chemistry A116
LUMSDAINE, A. A. AND GLASER, ROBERT, *Editors.* Teaching machines and programmed learning 275

MAHR, CARL. Anorganisches grund praktikum A916
MALLETTE, M. FRANK, ALTHOUSE, PAUL M., AND CLAGETT, CARL O. Biochemistry of plants and animals A282
MARKLEY, KLARE S., *Editor.* Fatty acids: Their chemistry, properties, production, and uses. Part 1 ..... A852
MARTIN, ALFRED N. Physical pharmacy .. A121
MARTINEZ, FRANCISCO B. AND BOUZA, ANTONIO. Aplicaciones analíticas del AEDT y similares 274
MASING, G. (ROGERS, B. A., *Translator*). Ternary systems A848
MASON, HOWARD S.—*See* FLORKIN, MARCEL
MELANDER, LARS. Isotope effects on reaction rates 330
MEYER, LILLIAN HOAGLAND. Food chemistry A202
MITCHELL, JOHN JR., KOLTHOFF, I. M., PROSKAUER, E. S., AND WEISSBERGER, A., *Editors.* Organic analysis. Volume 4 ..... A638
MORTON, MAURICE, *Editor.* Introduction to rubber technology 104
MOSER, C.—*See* DAUDEL, R.
MÜLLER, EUGENE, *Editor.* Methoden der organischen chemie. Vol. 5, Part 4: Halogenverbindungen A194
MUNZER, MARTHA E. AND BRANDWEIN, PAUL F. Teaching science through conservation 275
MYSELS, KAROL J. Introduction to colloid chemistry 50

NARAY-SZABO, ISTVAN. Anorganische chemie. Vol. 1 380
NERVIK, W. E.—*See* STEVENSON, P. C.
NEWMAN, MELVIN S., *Editor.* Organic syntheses. Vol. 40 ..... A444

O'BRIEN, RICHARD D. Toxic phosphorus esters 380
ONGLEY, P. A. Tutorial questions in organic chemistry A272
ORDWAY, RICHARD J.—*See* ALLEN, DONALD S.
OPOL, ARTHUR, *Editor.* The dispensatory of the United States of America. New drug development volume A926

Othmer, Donald F.—*See* Kirk, Raymond E.
Overman, Ralph T., and Clark, Herbert M. Radioisotope techniques..... A436

Partington, J. R. A history of Greek fire and gunpowder..... 430
A short history of chemistry..... A374
Pascal, Paul, *Editor*. Nouveau traité de chimie minérale. Vol. 7..... A116
—Vol. 15..... 331
Paterson, D. A., *Translator*. Instability constants of complex compounds..... 157
Pearson, R. G.—*See* Frost, A. A.
Perry, James W. Scientific Russian..... 535
Pfeiffer, H. G.—*See* Liebhafsky, H. A.
Pietsch, E. H. E., *Editor*. Gmelins handbuch der anorganischen chemie. 8th ed.
System 15: Silicon, part C..... A432
System 33: Cadmium supplement..... A372
Plane, Robert A.—*See* Sienko, Michell J.
Polianskii, N. G.—*See* Busev, A. I.
Pollack, Philip. Careers and opportunities in science..... A636
Potter, Van R. Nucleic acid outlines. Vol. 1..... A432
Poulik, M. D.—*See* Lewis, Lena A.
Proskauer, E. S.—*See* Mitchell, John, Jr.
Pyke, Magnus. About chemistry..... 379

Rabinowitch, Eugene—*See* Heidt, Lawrence J.
Radin, Norman, *Editor*. Radioisotope experiments for the chemistry curriculum..... 379
Raphael, Ralph A., Taylor, E. C., and Wynberg, Hans, *Editors*. Advances in organic chemistry: Methods and results. Vols. 1 and 2..... A438
Reid, Charles E. Principles of chemical thermodynamics..... 50
Reilley, Charles N., and Sawyer, Donald T. Experiments for instrumental methods..... 481
Roberts, J. K. Heat and thermodynamics 158
Robinson, P. L.—*See* Heslop, R. B.
Rochow, Eugene G., *Editor*. Inorganic syntheses. Volume 6..... A552
Rodd, E. H., *Editor*. Chemistry of carbon compounds. Volume 4. Part C: Heterocyclic compounds..... 588
Rogers, B. A.—*See* Masing, G.
Romer, Alfred. The restless atom. Vol. 12 of Science study series..... 588
Rosen, Milton J. and Goldsmith, Henry A. Chemical analysis. Vol. 12: Systematic analysis of surface-active agents..... A52
Royal Institute of Chemistry, London, England. Monographs for teachers... 537

Sachtleben, Rudolf, and Hermann, Armin. Von der Alchemie zur Grossynthese. Grosse Chemiker..... 380
Sanderson, R. T. Chemical periodicity.. 274
Sawyer, Donald T.—*See* Reilley, Charles N.
Schneer, Cecil J. The search for order.. A108
Schwenck, J. Rae—*See* Goostray, Stella
Seligson, David, *Editor*. Standard methods of clinical chemistry. Vol. 3..... 639
Seymour, Keith M. Organic chemistry.. 482
Sharpe, A. G.—*See* Stacey, M.
Sienko, Michell J., and Plane, Robert A. Chemistry..... 585
Simonds, Herbert R. Source book of the new plastic. Vol. 2..... A855
Simons, Joseph H. A structure of science. 276
Simpson, Stephen G.—*See* Hamilton, Leicester F.
Sisler, Harry H., VanderWerf, Calvin A., and Davidson, Arthur W. College chemistry: A systematic approach. A554
Smales, A. A., and Wagner, L. R., *Editors*. Methods in geochemistry..... A270
Smith, Cyril Stanley. A history of metallography..... 482
Solberg, Harry L., Cromer, Orville C., and Spalding, Albert R. Thermal engineering..... A204
Sorenson, Wayne, and Campbell, Tod W. Preparative methods of polymer chemistry..... A840
Spalding, Albert R.—*See* Solberg, Harry L.
Speakman, J. C.—*See* Brand, J. C. D.
Stacey, M., and Barker, S. A. Polysaccharides of micro-organisms..... 332
—Tatlow, J. C., and Sharpe, A. G., *Editors*. Advances in fluorine chemistry. Vol. 1..... A270
Standen, Anthony, *Editor*, Second supplement vol. of Encyclopedia of Chemical Technology (Edited by Kirk, R. E., and Othmer, D. F.)..... A370
Stanley, Wendell M., and Valens, Evans G. Viruses and the nature of life..... 535
Stevenson, P. C., and Nervik, W. E. The radiochemistry of the rare earths, scandium, yttrium, and actinium..... A768
Stewart, C. P., and Stolman, A., *Editors*. Toxicology: Mechanisms and analytical methods..... A923
Stewart, Warren E.—*See* Bird, R. Byron
Stickler, William C. General organic chemistry..... 534
Stolman, A.—*See* Stewart, C. P.
Strauss, Bernard S. An outline of chemical genetics..... A196
Stuart, A.—*See* Hartshorne, N. H.
Swallow, A. J. Radiation chemistry of organic compounds..... 640
Szent-Györgyi, Albert. Introduction to a submolecular biology..... 276

Tatlow, J. C.—*See* Stacey, M.
Taylor, E. C.—*See* Raphael, Ralph A.
Taylor, S. R.—*See* Ahrens, L. H.
Teuscher, H., Adler, R., and Seaton, Jerome P. The soil and its fertility.. A286
Theilheimer, W. Synthetic methods of organic chemistry. Vol. 14..... 330
Timmermans, Jean. The physico-chemical constants of binary systems in concentrated solutions. Vol. 4: Systems with inorganic plus organic or inorganic compounds (excepting metallic derivatives)..... A644
Tobolsky, A. V. Properties and structure of polymers..... A922
Trautmann, S.—*See* Ambard, L.
Tyree, S. Young, Jr., and Knox, Kerro. Textbook of inorganic chemistry..... 481

Van Orden, Harris O.—*See* Lee, Garth L.
VanderWerf, Calvin A.—*See* Sisler, Harry H.
Vasil'ev, V. P.—*See* Yatsimirskii, K. B.
Valens, Evans G.—*See* Stanley, Wendell M.
Vick, Maurice M.—*See* West, Philip W.
Vickery, R. C. Analytical chemistry of the rare earths..... A768
—The chemistry of yttrium and scandium..... A268
von Loesecke, Harry—*See* Harris, Robert S.

Wagner, L. R.—*See* Smales, A. A.
Watkins, Sidney B.—*See* Cremer, Herbert W.
Weissberger, Arnold, *Editor*. Physical methods of organic chemistry. Part 2. 222
Parts 3 and 4..... 432
—*See* Mitchell, John Jr.
Weisz, Herbert. Microanalysis by the ring oven technique..... 586
West, Philip W., and Vick, Maurice M. Qualitative analysis and analytical chemical separations..... A642
Wheeler, T. S.—*See* Gowan, J. E.
Wheland, G. W. Advanced organic chemistry..... 221
Whipple, Omer K. Chemical properties and identification of ions: An introduction to semi-micro qualitative analysis..... A920
Wiberg, Kenneth B. Laboratory technique in organic chemistry..... A192
Wilson, Cecil L., and Wilson, David W. Comprehensive analytical chemistry. Volume 1B, Classical analysis..... A766
Wilson, David W.—*See* Wilson, Cecil L.
Winslow, E. H.—*See* Liebhafsky, H. A.
Wyckoff, W. G. Crystal structures. Supplement 5..... A770
Wynberg, Hans—*See* Raphael, Ralph A.

Yatsimirskii, K. B., and Vasil'ev, V. P. Instability constants of complex compounds..... 157
Young, G. J., *Editor*. Fuel cells..... 157
Young, Roland S., *Editor*. Cobalt: Its chemistry, metallurgy and uses..... A498

Zeiss, H., *Editor*. Organometallic chemistry..... 587
Zeldovich, Ia. B., and Kompaneets, A. S. Theory of detonation..... 330
Zemany, P. D.—*See* Liebhafsky, H. A.

LETTERS TO THE EDITOR

Archer, R. D..... 638
Baker, Wilbur L..... 533
Battino, Rubin..... 102
Bieber, Theodore I..... 554
Broadbank, R. W. C., and Harding, R. D. 329
Cohen, Irwin..... 554
Crawford, Cratton M..... 637
Csanyi, Laszló J..... 155
Foy, John R..... 554
Harding, R. D.—*See* Broadbank, R. W. C.
Jellinek, H. H. G..... 378
Laughton, P. M..... 378
Mowery, Dwight F., Jr..... 329
Sampey, John R..... 102
Seymour, Raymond B..... 638
Smith, W. B..... 638
Taylor, D. A. H..... 102

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"This is a book which treats the fundamentals adequately and the applications superbly. Problems at the end of each chapter appear sensible and useful. Most of them give the impression of having been used, not just thrown in as an afterthought." (Jl. American Chemical Society)

This text places emphasis on recent advances, and thus more traditional material is treated in a rather condensed manner. For the undergraduate and graduate-level course, this book can be recommended enthusiastically.

508 pp, 97 illus, 1960—$9.75

CHEMICAL INSTRUMENTATION: A Systematic Approach to Instrumental Analysis

By Howard A. Strobel, *Duke University*

This text for a course of up to two semesters offers a systematic approach to the basic principles common to all methods and types of instruments and maintains a balance between physical theory and design on one hand, and chemical procedure and theory on the other. In each chapter are extensive references, plus well-chosen questions and problems dealing with the material of the chapter.

"This book is a refreshing departure from the usual loosely organized texts on the subject in that it treats instrumentation as a science in itself built on the foundations of physics, physical chemistry, and engineering. Amply illustrated with well-conceived figures." (Talanta)

653 pp, 373 illus, 1960—$9.75

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BOOK REVIEWS

Anorganisches Grundpraktikum

Carl Mahr, University of Marburg, Germany. 2nd ed. Verlag Chemie, GMBH, Weinheim/Bergstr., 1961. xiv + 421 pp. Figs. and tables. 15.5 × 23 cm. Paperbound, $7, Hard cover, $7.25.

This laboratory manual for first year chemistry in German universities is a continuation of Professor Mahr's attempts to introduce a change in the teaching of laboratory practice. The purely qualitative aspects have been de-emphasized, modern advances in techniques and measurements have been introduced. The author hopes that this will lead to shortening of the time required for the fulfillment of the degree requirements.

The experiments are grouped in sections designed to take about 3 to 4 hours of laboratory work. Each of these describes experiments demonstrating a particular technique.

The laboratory course is divided into three main parts, the first one pertaining to Introductory Practices, the second to Reactions of the Elements and the third to Qualitative Analysis.

The first part of the Introductory Practice covers techniques and measurements; it includes experiments on heating, glass blowing, weighing, volume measurements, miscible and immiscible solvent solute systems, etc. It is pretty much similar to the introductory experiments, although a greater variety in the various types is given. The second part demonstrates techniques used in the separation of substances such as filtration, centrifugation, differential solubilities, distillation, sublimation, etc. The third part of this section pertains to the application of the fundamental laws of chemistry to aqueous solutions. Among the various topics discussed and demonstrated are measurements of conductivity, ionic mobility, *p*H, ionization constant, buffer solutions, solvation and complex ion formation, solubility products, red-ox potential and electrolysis and reaction rates and reaction kinetics. Many interesting experiments have been devised in most cases with very simple equipment.

Twenty-four half days of laboratory work are necessary for these experiments which would give six weeks with two full days per week.

The second main part covers in about 200 experiments the reactions and qualitative determinations of the metals and nonmetals. Techniques and principles demonstrated in the first part are applied.

The first section of this part deals with the reactions and identifications of elements and compounds of Group VII-B through III-B. Preparations of the elements and their compounds are included. A few experiments of this section are of the type given in American freshman laboratory manuals. A large variety of experiments involve most of the usual compounds of the groups. Many of these experiments are challenging to first

(*Continued on page A918*)

From *theory* to *technique*— Macmillan texts are first in the field of *Chemistry*

PRINCIPLES AND PRACTICE OF ORGANIC CHEMISTRY

By Frederick G. Bordwell, Northwestern University

Here is the functional textbook that Chemistry instructors have been waiting for. Professor Bordwell, after nine years of classroom testing, has achieved total integration of functional classes by means of an organization based on structure and mechanistic reaction type. Three-dimensional structure receives particular attention. This book will prove a valuable addition to any chemistry library, in addition to being one of the most modern and effective texts available for classroom use.

April, 1962

ORGANIC CHEMISTRY LABORATORY OPERATIONS

By William B. Renfrow and Peter J. Hawkins, both of Oberlin College

This paperbound collection of experiments in organic chemistry stresses modern research methods. The exercises illustrate the techniques and basic principles of laboratory work, and are designed to accompany any standard text in the field. Qualitative and physical organic chemistry, and the design of experimental procedures receive particular attention. There is more material on qualitative organic analysis than in any comparable manual. The experiments have all been thoroughly tested under actual teaching conditions.

April, 1962

SEMIMICRO LABORATORY EXERCISES IN GENERAL CHEMISTRY, Third Edition

By J. Austin Burrows and Paul Arthur, both of Oklahoma State University, and Otto M. Smith, Kansas State Teacher's College

This outstanding laboratory manual for first year chemistry courses utilizes the more efficient and economical semimicro techniques for both quantitative and descriptive experiments. There are experiments in radioisotopes and modern applications of ion exchange, resins, paper chromatography, determination of ionization constants, and argentimetric titrations. Each experiment is accompanied by carefully prepared practice problems, tables of essential information and materials, and a complete list of reagents and necessary apparatus. The experiments can be used for both two and three-hour laboratory periods.

February, 1962

LABORATORY PRACTICE OF ORGANIC CHEMISTRY, Fourth Edition

By G. Ross Robertson and Thomas Jacobs, both of the University of California (Los Angeles)

The distinctive feature of this work is its extensive but informal treatment of the principles underlying laboratory practice. It encourages independent scientific thinking by emphasizing analytical experimentation and the preparation of chemical products in finished form. A "Library Problem" will assist the student in becoming aware of the writing skills necessary in the study of chemistry. The "Special Experiments" have been expanded, providing significant special problems for the second semester of a year course.

April, 1962

TEXTBOOK OF INORGANIC CHEMISTRY

By S. Young Tyree, University of North Carolina, and Kerro Knox, Bell Telephone Laboratories

Designed expressly for beginning courses in inorganic chemistry, this text requires no previous background in physical chemistry. Its overall view of the field highlights patterns of behavior among the elements. The material is organized according to groups of elements in the periodic table. For each element, the authors outline occurrence, history, preparation, physical properties, chemical properties and compounds. The new systematic nomenclature is employed throughout the text.

1961, 434 pages, $7.00

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year students such as those studying reaction kinetics, determination of coordination numbers, polymerization of phosphates, adsorption on Kieselguhr, Base-Antibase reactions. A few new techniques are introduced (such as paper chromatography) at appropriate places. The experiments in Group IV-b are restricted to those compounds considered primarily inorganic.

In the second section of this part the preparation and chemical properties of the metals and their compounds are combined with traditional H_2S separation scheme. New techniques such as Ion Exchange, Complexometry, etc., are presented for those compounds where their application is most appropriate. The author states that the purpose of the H_2S scheme is not to identify the components, but to separate them as pure compounds. Although this is stretching the point, the author does teach the student techniques necessary for success in trace analysis, radiochemical separations and preparation of pure compounds.

The last main part is devoted to qualitative analysis by micro and semimicro techniques. Its first section demonstrates the use of specific organic and inorganic reagents for metal ions and includes an introduction to spot plate techniques. The various types of reactions—salts, redox, complexation, etc., are described.

This is followed by an analytical scheme in an excellent condensed form. After a series of preliminary test-flame heating, the group separation involves among others the HNO_3 insoluble, HCl, reduction, thioacetamide, sulfate groups. The cations are identified by specific reagents, a short section on anions and insoluble substance identification is included. Short introductions to microscopic, trace and paper chromatic separation make up the rest of this part.

The author purposely does not discuss much of the theoretical aspects, but gives excellent references for these. The book should be of interest to American chemists because it demonstrates that German chemistry professors realize that changes in their beginning chemistry curriculum are long overdue. The reviewer also has found some of the sections of this book quite helpful.

HANS B. JONASSEN
Tulane University
New Orleans, Louisiana

Electronics for Spectroscopists

Edited by *C. G. Cannon*, British Nylon Spinners Ltd., Pontypool, England. Interscience Publishers, Inc., New York, 1960. viii + 333 pp. Figs. and tables. 15 × 22 cm. $9.50.

"Electronics for Spectroscopists" represents the proceedings of the 1949 and 1950 summer school courses organized by the British Photoelectric Spectrometry Group and the Electronics Department of Southampton University. There is a suggestion of inclusiveness in the title, but emission spectroscopists may be disappointed since their endeavors receive little direct attention except in portions of one chapter. Indeed, apparently because of the interests of many who were involved in its preparation, a large part of the book is directed toward those for whom a major concern is infrared spectrometry.

On the dust cover appears this statement: "This book aims at providing a complete course for spectroscopists who wish to learn something about electronics or to brush up their knowledge on the subject." For those who find themselves in the "brush up" category and who wish to become more thoroughly informed about specific topics, there will probably be found much that is helpful and satisfying. However, he who would "learn something about electronics" should first look elsewhere before he will be able to make effective use of the material presented here. Three authors contributed to the first 8 chapters whose topics include alternating current theory, amplifiers, power supplies, and noise. The approach is largely mathematical and would seem to presume some prior knowledge of basic electronics and perhaps a bit of circuit theory. Relatively little appears that could not be found in many other reference works and texts on electronics, but there is an admitted advantage in having this material collected and presented adjacent to other topics of interest to those who make use of the techniques of absorption spectros-

(*Continued on page A920*)

QUALITATIVE ELEMENTAL ANALYSIS

ERNEST H. SWIFT and W. P. SCHAEFER
California Institute of Technology

is a book for freshman and sophomore laboratory courses in qualitative chemistry. It employs the new system of analysis described by the authors in this issue of *Journal of Chemical Education* in the article entitled "A System of Qualitative Elemental Analysis for Instructional Purposes." The system embraces some thirty-two elements, which are divided into the Basic, Amphoteric, and Acidic Groups according to their acid- and base-forming properties. Because these properties can be readily correlated with the electronic structure and periodicity of their elements, this division offers distinct instructional advantages over the major group separations of conventional qualitative schemes. The system has been used for the past eight years with undergraduates at the California Institute of Technology and the University of California, Los Angeles.

1961, approx. 480 pages, $6.75

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☐ INTRODUCTION TO CHEMICAL INSTRUMENTATION

By EDWARD J. BAIR, Indiana University. McGraw-Hill Series in Advanced Chemistry. *Available January, 1962*

An introduction for the experimental chemist to a detailed understanding of the design and operation of chemical instruments. Emphasis is on the flow of signals and the information they represent. Designed for graduate students and research workers, the book covers a representative collection of topics which provide insight into new advances in instrumentation as they appear in chemical literature and into the art of designing instruments for new applications as well.

☐ STATISTICAL MECHANICS

By NORMAN DAVIDSON, California Institute of Technology. McGraw-Hill Series in Advanced Chemistry. *Available January, 1962.*

A text designed for a full year's course in statistical mechanics. The author helps the student gain control of this valuable tool of investigation, its techniques, and its limitations. The treatment is clear, readable and logical, but sufficiently rigorous for introductory or intermediate graduate courses. Careful attention is paid to detail and to mathematical development. A logical development from elementary to advanced theories.

☐ PHYSICAL CHEMISTRY

By GEORGE H. DUFFEY, South Dakota State College. Available January, 1962

This text has been prepared as an introduction to physical chemistry for students who have had calculus and have followed the modern chemical-bond approach in their introductory courses. The theoretical side of physical chemistry is emphasized and only enough descriptive and experimental material is presented to support the arguments and to form exercises in applying the theory.

☐ STEREOCHEMISTRY OF CARBON COMPOUNDS

By ERNEST L. ELIEL, University of Notre Dame. McGraw-Hill Series in Advanced Chemistry. *Available April, 1962.*

Using a sound theoretical approach based on the thermodynamic and kinetic concepts wherever possible, the book develops the essential concepts of stereochemistry on a level appropriate for students at the advanced undergraduate or graduate level. It will be of interest to organic chemists, biochemists, and physical chemists.

☐ NONEQUILIBRIUM THERMODYNAMICS: A Phenomenological Theory of Irreversible Processes in Fluid Systems

By DONALD D. FITTS, University of Pennsylvania. McGraw-Hill Series in Advanced Chemistry. *Available March, 1962.*

The purpose of this unique, advanced graduate textbook is to develop the macroscopic theory for transport processes in fluids—gases, gas mixtures, pure liquids and solutions. It develops the theory from clearly stated postulates and explores the consequences of the theory. The approach is analytical; the coverage is limited to the development of theory with some discussion and analysis of experimental results. Included is significant work which appeared late in 1960.

☐ THEORY OF MOLECULAR EXCITONS

By A. S. DAVYDOV. Translated from the first Russian edition by MICHAEL KASHA, Florida State University; and MAX OPPENHEIMER, JR., State University of Iowa. Available March, 1962.

Translation of a rare Russian book originally published in 1951 under the title, THEORY OF THE ABSORPTION OF LIGHT IN MOLECULAR CRYSTALS, by A. S. Davydov of the Ukranian Academy of Science. The author stresses the resonance interaction of weakly coupled molecular systems. To bring this edition up to date the translators have included two appendices which cover the principal literature on related topics through July, 1959.

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BOOK REVIEWS

copy. Since 10 years elapsed between the initial presentation of the unedited lectures and their appearance in bound form, it is unfortunate that certain chapters could not have been brought up to date in their literature coverage generally, and with some mention of solid-state electronic devices specifically.

A certain amount of useful information is to be found in the chapters entitled The Application of Semiconductors to the Detection of Radiation, Detector and Amplifier Systems in Infrared Spectrometry, Photoelectric Devices and Their Application in the Visible and Ultraviolet Regions, and Some Optical Problems. Two additional chapters are rather specialized: The Presentation of Infrared Spectra on the Cathode Ray Tube, with particular emphasis here being placed on the use of this technique in studying the kinetics of rapid reactions, and Polarized-Beam Techniques in Infrared Spectroscopy and their value, at least in simple cases, in determining approximate bond directions in a molecule.

Each of these six chapters in the latter half of the book is written by a different authority. The disadvantage of multiple authorship is at times apparent in the lack of uniformity of writing style, and in the scope and level of difficulty of the various offerings. In surveying the chapter on Polarized-Beam Techniques the reviewer found himself wishing that the extremely lucid manner of presentation shown there had extended to certain other portions of the book.

To continue in this vein might result in undue captiousness and convey a somewhat misleading impression. "Electronics for Spectroscopists" should prove valuable as a reference work for the serious spectroscopist who wishes to learn more about the topics that are included, and certainly the somewhat less expert spectrometer user who is able to master the material presented will have reasonably thorough and useful grounding in electronics, for application not only to his work in absorption spectroscopy but possibly to other research problems as well. Further, those in education who are responsible for incorporating into the curriculum basic instruction in spectroscopy should find considerable information of help to them.

H. W. SAFFORD
University of Pittsburgh
Pittsburgh, Pennsylvania

Chemical Properties and Identification of Ions: An Introduction to Semimicro Qualitative Analysis

Omer K. Whipple, California State Polytechnic College, San Luis Obispo. Wadsworth Publishing Co., Inc., San Francisco, California, 1961. x + 310 pp. Figs. and tables. 15 × 20 cm. Paperbound. $3.95.

In the preface of this little laboratory book the statement is made that "It is designed particularly for use in the second

(*Continued on page A922*)

JUST PUBLISHED

. . . a general chemistry laboratory manual designed to minimize the "cookbook" nature of the first year chemistry laboratory.

INVESTIGATIONS IN CHEMISTRY

by Jay E. Taylor, *Kent State University*, John R. Demuth, *University of Nebraska* and George L. Bush, *Kent State University*

The unique and most outstanding feature of this manual is the investigative approach. Numerous suggestions give the student excellent opportunity for supervised independent work. Included are more experiments of a quantitative nature than normally found in general chemistry manuals. Experiments dealing with chemical kinetics, equilibrium, solubility product, voltaic cells, conductivity of solutions are original in design and emphasize a high level approach to the understanding of underlying principles. A formal scheme of qualitative analysis outlines laboratory work for those colleges which devote all or part of the second semester of general chemistry to this area of instruction. Suggestions are given for individual student development of a scheme of qualitative analysis. Ideas for a special student project are presented. *1961, 250 pages, $4.25.*

. . . uses quantities of one-tenth of those commonly applied.

ORGANIC CHEMISTRY—LABORATORY EXERCISES

by James M. Sugihara, *University of Utah*

This manual meets the needs of chemistry majors, engineers and pre-medical students taking a beginning organic chemistry course. Experiments are described using one-tenth of those commonly applied . . . using conventional equipment but smaller in size. Among the advantages gained are: greater safety in student laboratories, lower replacement cost of broken equipment, use of more expensive chemicals, less time required for completion of experiments and less space required. *1961, 128 pages, $4.00.*

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ABSORPTION SPECTROSCOPY

By ROBERT P. BAUMAN, *Polytechnic Institute of Brooklyn.* A comprehensive introduction to postwar theory and practice in absorption spectroscopy, this book covers ultraviolet-visible, infrared, and Raman spectroscopic methods and their applications in qualitative and quantitative analysis and in the determination of molecular structure. It will enable the student with no prior experience in the subject to take advantage of the many valuable specialized monographs and current research articles in the field. The author begins by developing the theory underlying interpretation of spectra and pertinent aspects of classical and quantum mechanics. He then goes on to discuss qualitative analysis by means of electronic and vibrational spectra, quantitative analysis through ideal and non-ideal systems of one or many components, and molecular spectroscopy. Problems are included at the end of each chapter.

1962. Approx. 568 pages. Prob. $10.50

BASIC PRINCIPLES OF THE TRACER METHOD

By C. W. SHEPPARD, *University of Tennessee Medical Units.* This monograph brings together and formulates for the first time the basic principles of tracer kinetic theory and analysis. The author's aim is to show the student the scope and limitations of the tracer method—particularly, with respect to its applications in physics, chemistry, and physiology. He also discusses what may be expected to occur in a kinetic system following the introduction of labelled material into a part of it. Theoretical material is necessarily presented in mathematical terms, but in such a way that students without an extensive background in mathematics can follow the analysis. Special attention is given to modern methods of applied mathematics, including numerical analysis with digital computers and analog simulation procedures. A valuable and extensive bibliography appears at the end of the book.

1962. Approx. 275 pages. Prob. $8.00

DYNAMIC PHYSICAL CHEMISTRY

By J. ROSE, *Birkenhead Technical College, Great Britain.* Designed to serve as either an introduction or a reference to modern physical chemistry, this book is a clear, easy-to-follow guide to the many important recent advances in the field. One of the major features of the book is the attention it devotes to special subjects which are now becoming important, e.g., reactions in the solid state and at elevated temperatures, radiochemical kinetics, and the Principle of Caratheodory. In addition, there are extensive treatments of thermal kinetics, photochemistry, phase rule, and thermodynamics. Quantum theory is introduced by describing the experimental basis of the concept, and both the "old" and the "new" theories are discussed with special reference to the periodic classification and bonding.

1961. In press.

BIOCHEMICAL MECHANISMS

By LLOYD L. INGRAHAM, *University of California, Davis,* Biochemistry has now reached a stage of development that requires a knowledge of mechanistic chemical principles. Although there are several books which contain background material on these principles, this book is the first to fill the special needs of biochemists in this area. Part I of the book presents the general mechanistic principles of organic and inorganic chemistry that are of value in biochemistry. It includes a review of orbital and other fairly basic concepts in order to facilitate the discussion of mechanisms in the second section of the book. Part II treats specific biochemical mechanisms, and is divided into discussions of various types of reactions. The mechanism as known in solution chemistry is discussed in terms of principles that are applicable to the biochemical reaction. Next, the mechanisms of action of any model enzyme are described, and finally the enzymic reaction is compared with model reactions. *1962. Approx. 112 pages. Prob. $4.75*

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term of the first year chemistry course," and the book seems to meet this design adequately.

The first two chapters involve some discussion of chemical reactions, contrasting molecular with ionic reactions, and equation writing of various types with emphasis on those involving oxidation-reduction. Semimicro equipment, materials, and techniques are rather nicely discussed.

The anion groups are divided into three types, the chloride group, the sulfide group, and the nitrate group. There are 14 anions involved.

There is an adequate general discussion of the standard division of cation groups, i.e., sodium-calcium, iron-aluminum, copper-tin, and silver. A short coverage is given to the solvent action of water on electrolytes, but there is a good development of K_{sp} and the ion product. Many excellent problems illustrate the concepts involved.

In the experimental section on cations, the groups are somewhat abridged in the interest of economy of laboratory time. For instance, strontium is omitted from the sodium-calcium group, cobalt is omitted from the iron-aluminum group, and bismuth, arsenic, and mercury II are omitted from the copper-tin group. There is an adequate discussion of complex ions and instability constants again with appropriate examples of various calculations.

Each chapter is followed by a sufficient number of problems so that considerable selection by the teacher is available. Answers are given to the mathematical questions.

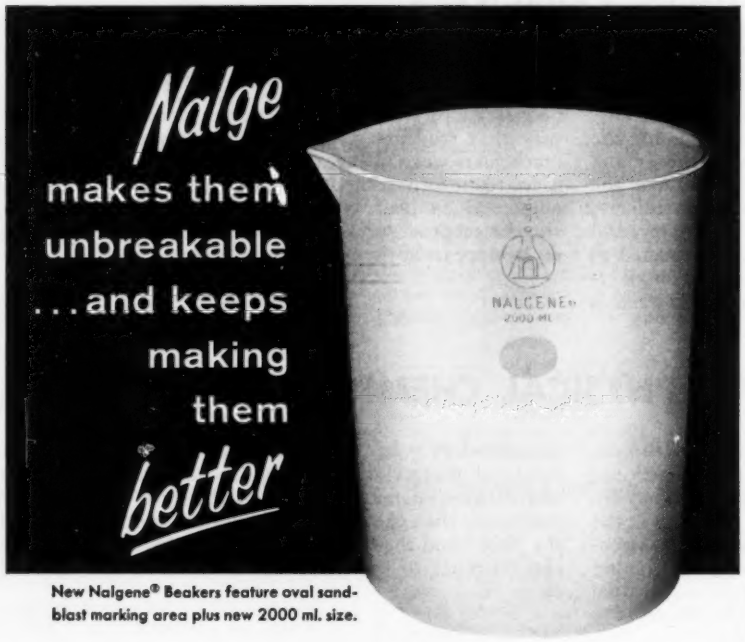
After the experimental work on each individual anion or cation is completed, directions and a known solution of the entire group is provided along with a well-done flow sheet for that particular group. After the student has reported his results on a specially designed report form for the known solution, he is given an unknown solution with instructions to devise his own method of analysis and to take advantage of any short cuts which he may deem advisable. Again a special report form is provided.

With the emphasis that the author puts on oxidation-reduction in a qualitative manner, it seems peculiar that no mention is made of oxidation potentials. Such an inclusion might enhance the understanding that the student could obtain from the use of this book.

In the various appendices there are given suggestions for recording laboratory observations, lists of student locker equipment, lists of student and general reagents, a time saving section on the preparation of solutions, directions for issuing and reporting unknown solutions, and a table of four place logarithms.

All in all, the book is well written, explicit, and useful in providing an adequate second semester laboratory experience for general chemistry students.

DONALD B. SUMMERS
Olin Mathieson Visiting
Chemistry Teacher
Alton Senior High School
Alton, Illinois



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Properties and Structure of Polymers

A. V. Tobolsky, Princeton University, Princeton, New Jersey. John Wiley & Sons, Inc., New York, 1960. x + 331 pp. Figs. and tables. 16 × 23.5 cm. $14.50.

This book provides information for the specialist or for the physical chemist or physicist who has an active interest in the specific concepts discussed. The subjects of elasticity and viscosity, polymer physics, linear viscoelasticity, viscoelastic behavior, chemical stress relaxation and polymerization equilibria are discussed and treated mathematically in an admirable manner. Several physical concepts are discussed briefly under the subject of "polymer physics." Among these are the conformation of a linear chain, amorphous and crystalline polymers, thermodynamics and kinetics of crystallization, the glass transition temperature, regions of viscoelastic behavior, modulus-temperature curves, flow viscosity of linear amorphous polymers, and the kinetic theory of rubber elasticity.

The book is written predominantly as a treatment of polymers having properties which permit them to fit into the area of elastomers, rather than from the standpoint of polymers in general. The major portion of the illustrative material is taken from that group which are predominantly useful as elastomers. While the author has given adequate reference material and, in many cases, as he points out in the preface, citation to the original experimental work has been made, many topics of extreme importance to properties and struc-

BOOK REVIEWS

tures of polymers have not been referred to at all, or have been mentioned only briefly. Among these are chemical bonds and molecular forces, infrared methods of determination of chemical structure of polymers, thermal stability of polymers, effects of degree of crystallinity and molecular weight on tensile strength, relationship of polymer properties for plastic, fiber or elastomer application, steric hindrance in polymer chains, the theory of gelation, properties of polyelectrolytes, properties of proteins, and cellulosic materials.

The author points out in the preface that "only selected topics could be treated in a book of modest size, and the choices were naturally guided by my own research experiences." His interests in viscoelasticity have predominated in his thinking, and many of the other physical concepts discussed have been used as supporting data for the major topic of viscoelastic behavior. The only criticism of this book can be that the title is too broad rather than that the subject matter treatment is inadequate. As pointed out above, the theoretical and mathematical treatment of the chosen subject matter have been done in an excellent manner.

GEORGE B. BUTLER
University of Florida
Gainesville

The Use of Organic Reagents in Inorganic Analysis

A. I. Busev and *N. G. Polianskii.* Translated from the Russian by *J. T. Greaves.* Pergamon Press, Inc., New York, 1960. viii + 76 pp. 14.5 × 22 cm. $3.50.

This short monograph, an English translation of the original Russian text, should prove quite useful to chemists in this country. The first section of this book treats the problems one encounters in the application of organic reagents. Short abstracts of theoretical studies of complex formation are presented in this section. The second portion of the text gives, according to a periodic group classification, methods of determining many of the elements using organic reagents.

Most of the references in this text cover the years from 1953 through 1955. This limitation prevents any critical treatment of organic reagents such as that presented in references by Welcher or Feigl. The translation is not well written, but nevertheless the material will be useful in the study of organic reagents.

An attractive feature of this text is the incorporation of material from Russian journals. The English translations of all the original articles are available from the publisher. These factors should enhance the usefulness of this monograph.

THEODORE R. WILLIAMS
College of Wooster
Wooster, Ohio

Toxicology: Mechanisms and Analytical Methods. Volume 1

Edited by *C P. Stewart,* University of Edinburgh, Scotland and *A. Stolman,* Connecticut State Department of Health, Hartford. Academic Press, Inc., New York, 1960. xvii + 774 pp. Figs. and tables. 16.5 × 23.5 cm. $22.

The scope of toxicology in this book is largely forensic; the mechanisms are to a large extent those of drugs, and the analytical methods relate to body tissues and excreta.

Toxicology, which in the older and stricter view might be considered as the science of poisons, rather today is in reality a science devoted to the determination for *any material* under what conditions it is either safe or beneficial. This concept is utilized in research in establishing limits of safety, control of the environment, the surveillance of man as related to industrial exposure, air and water pollution. This concept applies equally to the foods eaten by the general public, to drug action, to forensic medicine, etc. Since the present day pursuit of this field of knowledge, as many others, involves the use of many disciplines, it is reasonably understandable why no one author nor any group has attempted to provide a series of related comprehensive books pertaining to the toxicological facts, *per se,* as well as the knowledge and techniques as applied by the numerous disciplines. It is unfortunate that a book as well written and as authoritative in its particular aspect of toxicology unwittingly leaves the impression that its phase is either the only or the major portion of toxicology.

One will not find here elaboration of such basic toxicological concepts as dosage-response, tolerance, poison, safety, or hazard. Toxicological facts, *per se,* symptomatology and pathology are considered only slightly or not at all.

The authors correctly call attention to the fact that upon inhalation of a gas or vapor that the time required for saturation (equilibrium) of the body by a nonreactive material is inversely related to the solubility. However, they do not make it clear that the concentration in the blood at equilibrium is unrelated to solubility but rather related to the distribution coefficient between the blood and the alveolar air. Although valuable blood levels are related to clinical conditions, the concentrations inhaled and times to attain such levels generally are not reported.

This book explains well the principles of the method of analysis, although, at times the discussions are a little meager. For example, the infrared instrumentation presents an optical diagram of a typical spectrometer which has been supplanted by more up-to-date instruments. It is our opinion that section VII (page 338), Gas Chromatography, was sadly neglected. However, the other fields of interest and remaining discussions, while brief, may be adequate for a worker who wishes to familiarize himself with the general principles of analysis. The refer-

(*Continued on page A924*)

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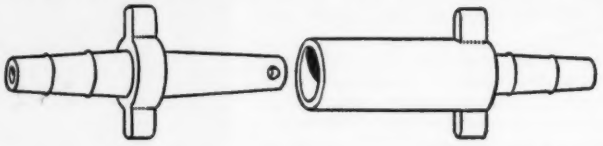

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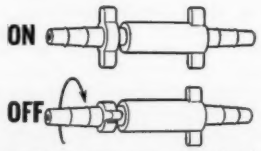

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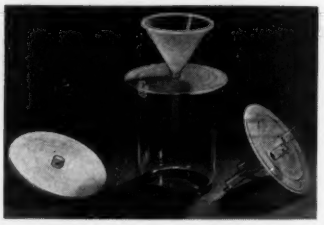

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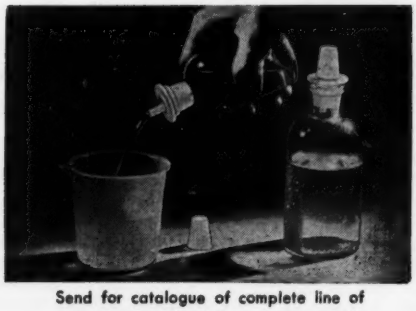

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Although it excludes many compounds from the sections relating to the fate of material, the book brings together considerable important information relating to fate of materials, with emphasis on drugs, on levels of compounds in blood, tissues, respired air, and urine. The fate of insecticides is given brief attention. The recent excellent series of studies by Gerade on the fate of aromatic hydrocarbons are conspicuous by their absence but this is probably due to time required to produce such a book.

In general, the editors have accomplished their purpose since it presents excellent material and references from the standpoint of forensic toxicology and toxicological analysis. We believe this book will become a valuable addition to the practicing professional toxicologists, and clinical chemists who are confronted with "what method of separation, of qualitative identification and estimation is best suited for the problem."

JOSEPH F. TREON and
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Progress in Inorganic Chemistry. Volume 2

Edited by *F. Albert Cotton*, Massachusetts Institue of Technology, Cambridge. Interscience Publishers, Inc., New York, 1960. x + 399 pp. Figs. and tables. 16 × 23.5 cm. $10.50.

The second volume in this series consists of seven chapters. These range in scope from strictly theoretical discussions to essentially descriptive summaries of subjects which are currently engaging the interests of inorganic chemists.

The first chapter, on "Radioactivation Analysis in Inorganic Chemistry" by J. W. Winchester, is concerned with problems such as chemical dating and terrestial composition. This review examines critically the reliability and sensitivity of several analytical methods, with particular emphasis on the excellence of that method based on neutron activation. Extensive tables are provided giving the abundance of elements in silicate rocks, etc. R. M. Diamond and D. C. Tuck, in a second chapter, present a comprehensive and critical review covering the "Extraction of Inorganic Compounds into Organic Solvents." In the opinion of this reviewer, the subject is presented in a most excellent fashion, both theoretically and topically. Obviously, the practical applications of this method are becoming increasingly more important. In a third chapter C. J. Ballhausen considers the "Intensities of Spectral Bands in Transition Metal Complexes." While the presentation is excellent, this reviewer feels that a more comprehensive treatment with

(*Continued on page A926*)

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(1) Ind. Eng. Chem. 26–653 (June, 1933)

(2) Ind. Eng. Chem. 25–1112 (Oct., 1933)

(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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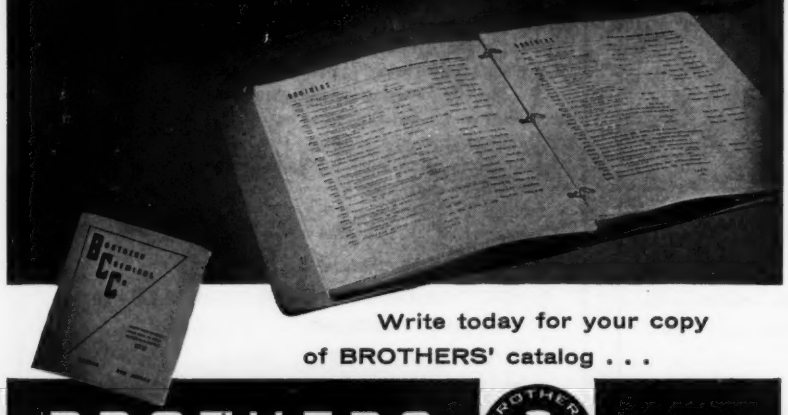

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The four remaining chapters are devoted, primarily, to descriptive chemistry, but a discussion of structures and the application of physical principles are not neglected. These are: "Halides and Oxyhalides of the Elements of Groups Vb and VIb" by John W. George; "Some Fluorine Compounds of the Transition Metals" by R. D. Peacock; "Unusual Oxidation States of Some Actinide and Lanthanide Elements" by L. B. Aeprey and B. B. Cunningham, and "Metal Alkoxides" by D. C. Bradley. The review on fluorine compounds by Peacock is notable in that it includes a section on preparative methods and techniques.

In view of the rapidly expanding areas in inorganic chemistry it is becoming increasingly apparent to all that it is impossible to keep abreast with modern developments by merely making a casual survey of the literature. Accordingly critical reviews such as these are becoming more and more important. All persons interested in developments in inorganic chemistry should have access to these collected works.

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Edited by *Arthur Osol*, Philadelphia College of Pharmacy and Science, Philadelphia, Pennsylvania and *Robertson Pratt*, University of California School of Pharmacy. J. B. Lippincott Co., Philadelphia, Pennsylvania, 1960. 1960 ed. vii + 240 pp. 18 × 26.5 cm. $9.

This volume is a welcome addition to the pharmacists' and chemists' shelf as well as to the teachers' library. The purpose of this volume is to bring the U.S.D. 25 up to date with respect to the U.S.P. XVI, N.F. XI, B.P. 1958, and the International Pharmacopoeia. In addition to containing 209 completely new monographs, the supplement also contains survey articles on various categories of medicinals such as Antibiotics, Antibiotics with Antitumor Activity, Hypoglycemic Sulfonylureas and Biguanides, Psychotherapeutic and Psychotomimetic drugs, and Saluretic Agents. Extensive information is also presented on the manufacture and standardization of antibiotics. The authors have continued to concentrate on the actions, uses, doses, and side effects of the drugs discussed.

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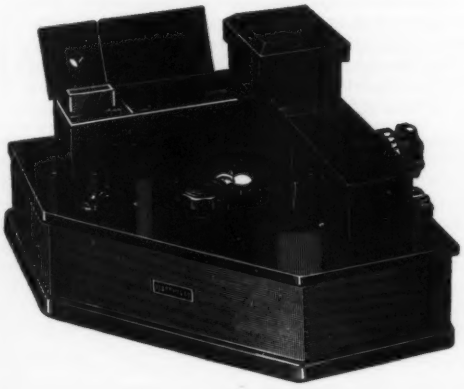

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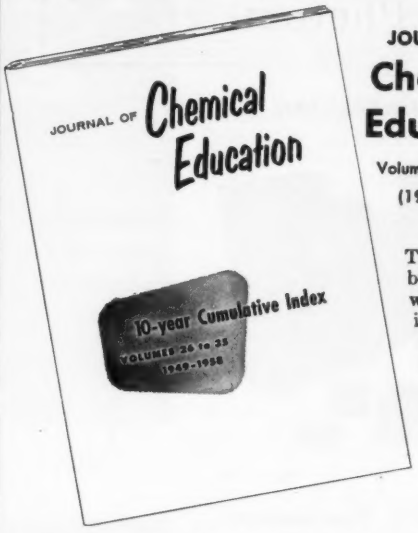

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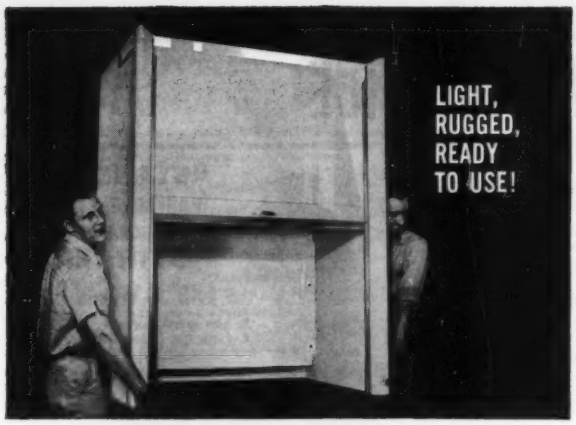

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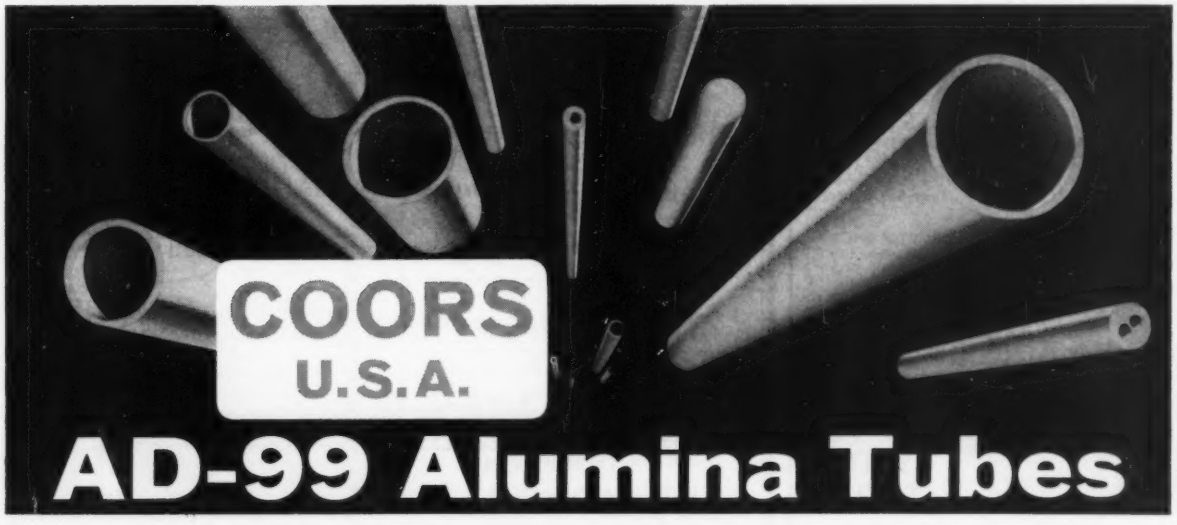

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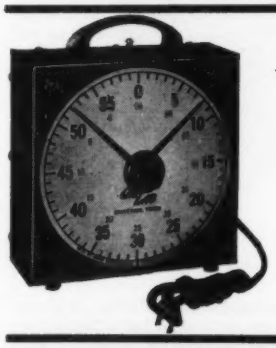
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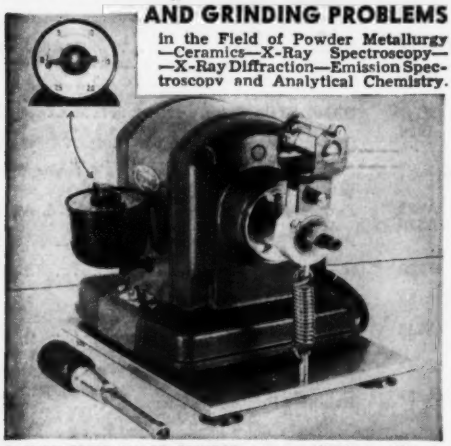

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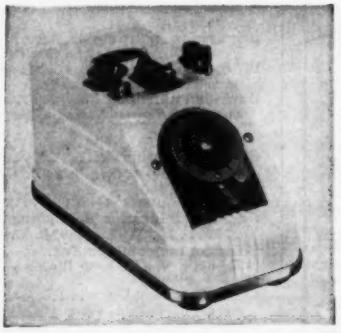

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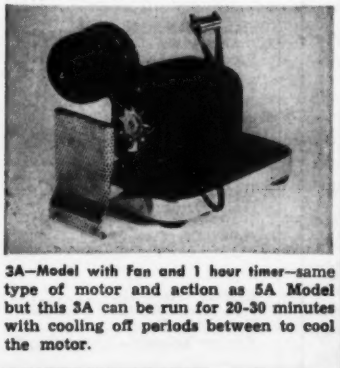

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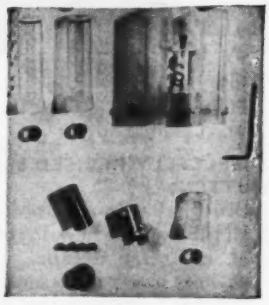

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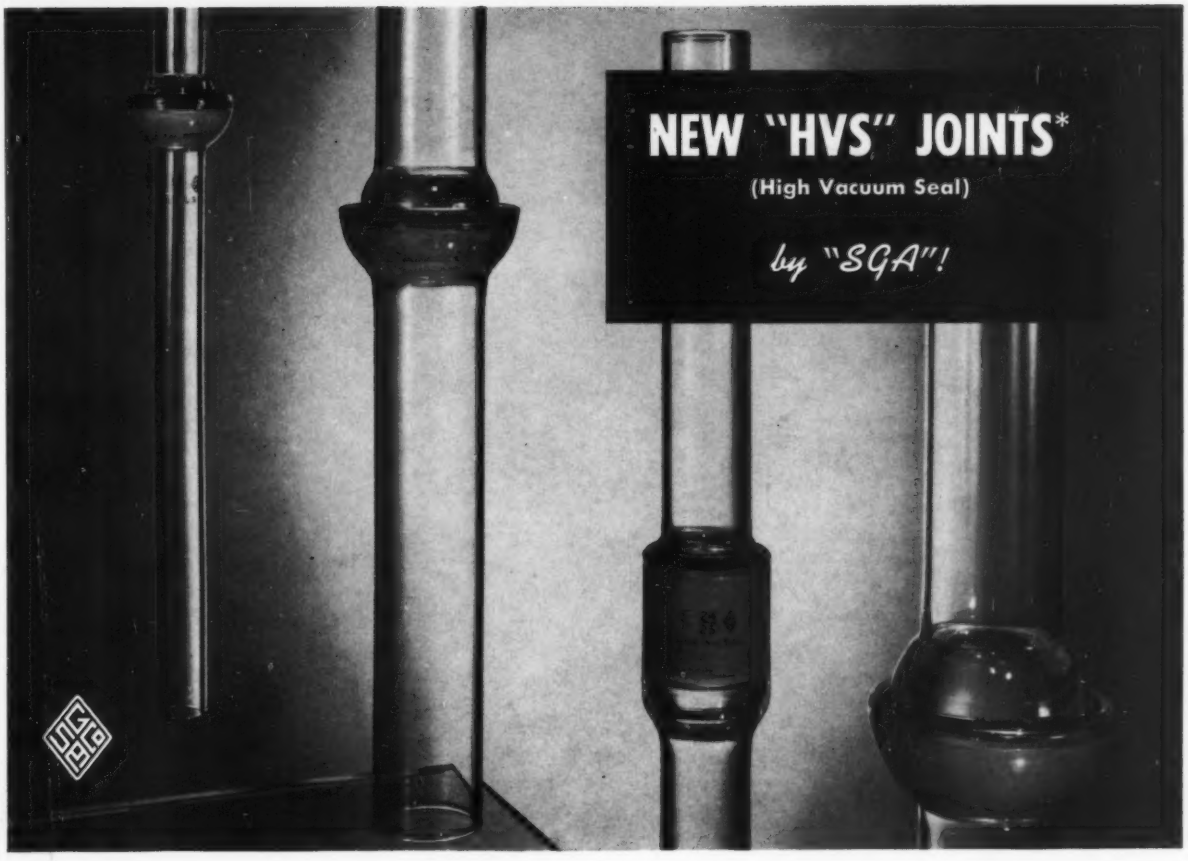

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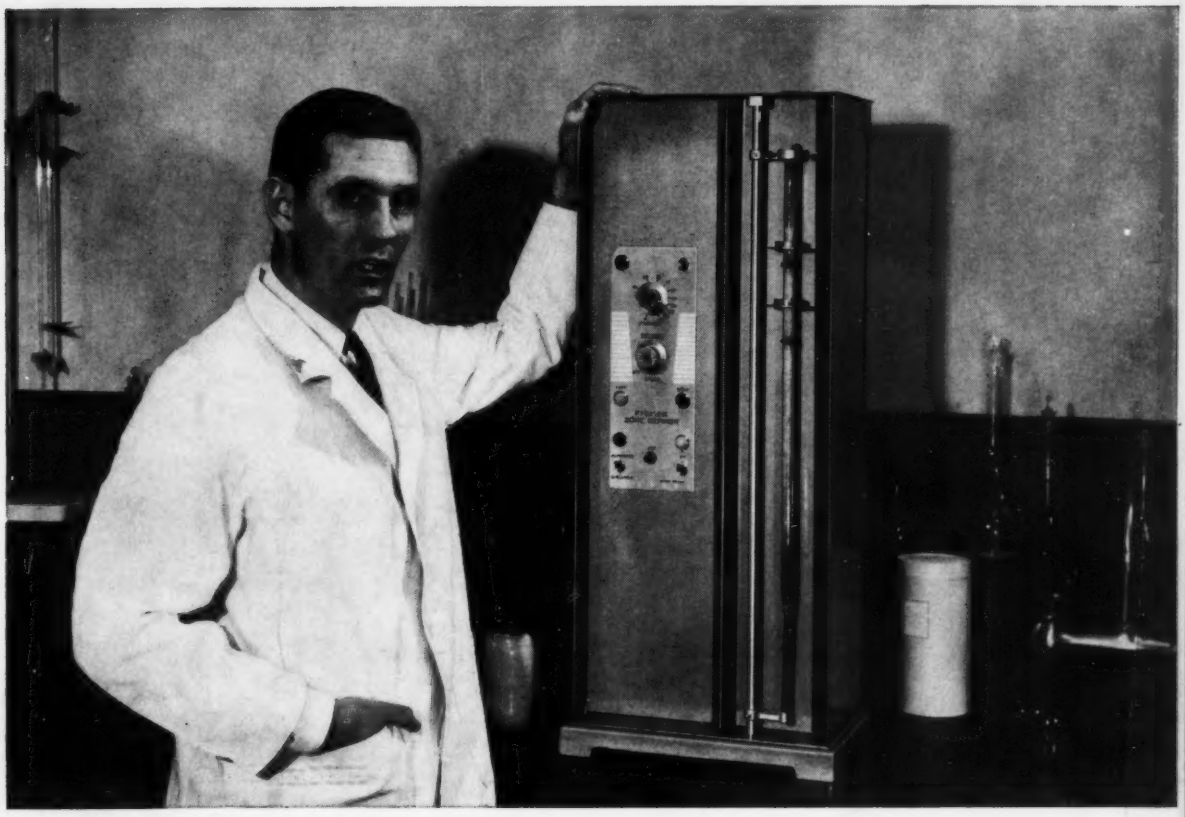

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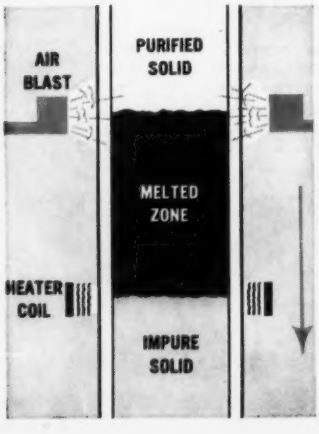

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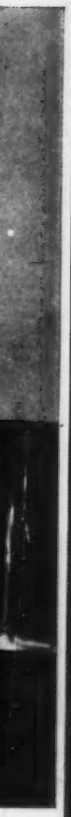
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